

Plasma Science and Technology

Room 201 ABCD W - Session PS-MoM

Advanced Logic and EUV Patterning

Moderators: John Arnold, IBM, Angelique Raley, TEL Technology Center, America, LLC

8:15am **PS-MoM-1 Current Status and Future Perspectives of Plasma-Induced Damage and its Characterization, Koji Eriguchi**, Kyoto University, Japan

INVITED

Defect generation during plasma processes (plasma-induced damage, PID) is a crucial challenge in electronic device design. Various techniques, including atomic layer etching and cryogenic processing, are believed to control PID. To ensure low PID, precise characterizations and a deeper understanding of the fundamental physics behind experimental observations are indispensable. This study reports recent progress in PID characterization and discusses the fundamental aspects as future perspectives.

PID is now evaluated by electrical methods [1]. With the introduction of low- k films, mechanical property changes by PID have become another focus. Recently, a cyclic nanoindentation (c-NI) method [2] revealed that SiN films degrade more significantly than SiO₂ films by PID, particularly in terms of contact stiffness evolution. Mechanical property changes by PID should be considered for various applications.

Impedance spectroscopy (IS) was proposed to assess various aspects of PID in SiN and SiO₂ films [3]. An IS-based method was applied to Si and InP [4], one of the promising compound semiconductors for next-generation devices. Capacitive and conductive components were found to depend on the incident species from plasma. The IS-based method identified differences in the energy profiles of defects created in Si and InP substrates.

PID is governed by indeterministic dynamics. Molecular dynamics simulations [5] revealed that PID is formed by stochastic straggling within the sidewalls of "fin" structures. An increase in junction current (~dark current in CMOS image sensors) was experimentally confirmed using device arrays [6], highlighting the need to incorporate stochastic mechanisms in designing plasma processes for 3D and ultimately scaled devices. Recently, the stochastic aspects of PID were modeled similarly to stock price predictions, revealing Poisson statistics in defect creation and fundamental PID variation [7].

PID is inherently unavoidable. A deeper understanding and advanced characterization techniques are indispensable. This study reports mechanical property degradation and new characterization methods. Stochastic mechanisms in 3D and ultimately scaled devices have been discussed. Exploring various techniques and predictive models will be essential for future PID design.

[1] K. Eriguchi, Jpn. J. Appl. Phys. **60**, 040101 (2021).

[2] T. Goya *et al.*, J. Phys. D **57**, 475202 (2024).

[3] T. Kuyama *et al.*, IEEE IRPS, 4B.4 (2021).

[4] T. Goya *et al.*, Jpn. J. Appl. Phys. **63**, 06SP04 (2024).

[5] K. Eriguchi *et al.*, Jpn. J. Appl. Phys. **53**, 03DE02 (2014).

[6] Y. Sato *et al.*, J. Vac. Sci. Technol. B **40**, 062209 (2022).

[7] K. Eriguchi and K. Urabe, Dry Process Symp., 17 (2023).

8:45am **PS-MoM-3 Direct Etching of Ru Pattern with Space Width of 10 nm and Less, Miyako Matsui**, Hitachi, Ltd., Japan; Kiyohiko Sato, Makoto Miura, Kenichi Kuwahara, Hitachi High-Tech Corp., Japan

With continuous device scaling, scaling of the metal pitch is continuously required using advanced patterning technologies such as extreme ultraviolet lithography. As such scaling continues, alternative metal interconnects are required to replace Cu. Ru is a candidate for an alternative interconnect material with metal pitches of 20 nm and beyond because a Ru interconnect can have a lower effective resistance than that of a Cu interconnect at such small pitches. Ru can be etched directly, which can lead to new scaling boosters such as semi-damascene patterning. In the Ru etching process, roughness or other damage should be suppressed to reduce interconnect resistance.

In our previous study, we investigated the mechanism generating roughness in Ru patterns with a 32-nm pitch by using Cl₂/O₂-based plasma generated by a microwave-ECR etching system. The Ru sidewall roughness was thought to be caused by non-volatile RuO_x and RuCl_y, which were non-

uniformly formed on the Ru sidewall. We found that the sidewall roughness of a Ru pattern strongly depends on the protection layers formed on the sidewall by adding passivation gas to the Cl₂/O₂-based plasma.

In this study, we investigated the etching mechanism of Ru patterns with a space width of 10 nm or less. The effect of space width on the etching performance and cross-sectional profiles of Ru patterns with a narrow space width was investigated. The space width of a Si₃N₄ mask pattern with a width of 14 nm was narrowed using an in-situ atomic-level deposition technique, which almost conformally deposited an atomic-level-thick SiO₂ layer on a Si₃N₄ mask pattern with a 32-nm pitch.

Etch stop occurred on the patterns when the space width of the mask pattern was narrower than 9 nm because the SiO₂/Si₃N₄ hard mask widened due to the redeposition of Si-containing by-products, and the redeposited by-products prevented ions from entering the space. When the ion energy was increased by applying a higher wafer bias power and the ion flux decreased by reducing the duty cycle of the wafer bias, Ru etching proceeded, but the Ru sidewall was laterally etched by radicals during the off-period of the wafer bias power. Ru sidewall protection becomes more important in preventing the lateral etching of Ru patterns with a narrow space because the ratio of radical to ion flux becomes large inside the narrow space.

We suggest adding a passivation gas to the Cl₂/O₂ plasma to form a protection layer on the sidewall of patterns with a space width of 10 nm or less. Sidewall roughness was also reduced because the Ru sidewall was protected from the etching by a uniform protection layer.

9:00am **PS-MoM-4 Study of Electron-Surface Interactions for Etching of Ruthenium with Chlorine and Oxygen, Michael Hinshelwood¹**, University of Maryland College Park; Hubertus Marbach, Michael Rimmel, Gerson Mette, Michael Budach, Daniel Rhinow, Klaus Edinger, Carl Zeiss SMT GmbH, Germany; Gottlieb S. Oehrlein, University of Maryland College Park

Ruthenium (Ru), a material used in semiconductor manufacturing for extreme ultraviolet (EUV) photomasks, is experiencing increasing attention as a material for capping layer and as potential adsorber in high NA lithography. Ru can be etched through the formation of volatile oxides and considerable research efforts have been put into optimizing etching processes. Y. Li *et al.* demonstrated that Ru can be rapidly etched by combining a low-energy electron beam (EB) with a flux of O₂/Cl₂-derived neutrals emanating from a remote plasma (RP) source [1]. This etching demonstrated a synergistic effect, with the remote plasma by itself causing a low level of etching, and the combination of EB and unexcited gas resulting in growth of a non-volatile compound. Here, we build upon that work, using in-situ ellipsometry to gain understanding on how the EB affects the surface etching reaction. By breaking up the exposure steps, subjecting the Ru surface to sequential EB and RP with either Cl₂ or O₂, we have found that the EB in conjunction with Cl₂ gas induces the uptake of Cl on the Ru surface, creating a chlorinated layer. The growth rate of this layer increases with increasing electron flux and energy, suggesting that electron-induced modification of the Ru surface is the source of the observed behavior. This chlorinated layer is selectively etched by O₂ plasma-derived neutrals, which otherwise results in non-volatile RuO₂ growth on the unmodified Ru. The etch rate of this modified layer increases with layer thickness, and is boosted by electron flux, suggesting that the electrons have multiple roles in this etching system. By isolating the effects of the EB on this etch mechanism of Ru, we obtained valuable information for the development of low-damage etch processes. Ellipsometric data and models of surface processes are complemented by X-ray photoelectron spectroscopy data.

This material is based upon work supported by Carl Zeiss SMT GmbH.

[1] Y. Li *et al.*, "Investigation of ruthenium etching induced by electron beam irradiation and O₂/Cl₂ remote plasma-based neutral fluxes: Mechanistic insights and etching model," J. Vac. Sci. Technol. A, vol. 43, no. 2, p. 023005, Feb. 2025, doi: 10.1116/6.0004219.

9:15am **PS-MoM-5 Highly Selective Isotropic etching of SiGe over Si via Pulsed RF Power in NF₃ Plasma, Geun Young Yeom, Hong Seong Gil, Woo Chang Park, Yun Jong Jang**, Sungkyunkwan University (SKKU), Republic of Korea

Highly selective isotropic dry etching of SiGe for Si is an important process for fabricating 3D-structured GAA-FETs or 3D DRAM. Si-Ge bonds, which are relatively weaker than Si-Si bonds, are more easily fluorinated by F-radicals and thus are etched as SiF₄ and GeF₄. However, because the high reactivity

of fluorine radicals makes it challenging to achieve damage-free SiGe etching using F-radical-based approach, there is a growing demand for the development of more highly selective etch processes.

In this study, we employed an ICP-type remote plasma with NF_3 gas to investigate how pulsed RF power can be used to control the etch species and thereby to improve etch characteristics in isotropic SiGe etching. By applying microsecond-scale on/off cycles to the RF power, we found that the etch rate of SiGe maintained (or increased) while that of Si decreased, significantly enhancing the overall etch selectivity. Additionally, we found that lower processing temperatures significantly enhanced the etch selectivity, primarily due to differences in activation energies between Si and SiGe under pulsed plasma conditions.

9:30am PS-MoM-6 Significance of the Impacts of Metal Oxide Resists (MORs) in Plasma Etch Processes, Adam Pranda, Steven Grzeskowiak, Yusuke Yoshida, Eric Liu, TEL Technology Center, America, LLC

One of the major developments in logic scaling has been the transition from 193nm deep ultraviolet (DUV) lithography to 13.5nm extreme ultraviolet (EUV) lithography. Historically, organic or chemically amplified resists (CARs) have been used for patterning because of sufficient radiation sensitivity, resolution, and etch resistance for enabling viable manufacturing flows with DUV lithography. However, the shift to EUV lithography presents numerous problems for CARs due to the reduced absorption of EUV light that among roughness and defectivity challenges also necessitates thinner resist thickness to achieve high resolution. For etch processes utilizing EUV CARs, this requires maintaining a sufficiently high etch selectivity to successfully transfer the pattern before integrity is lost. One approach to overcoming the challenges with CARs in EUV lithography has been the development of metal oxide resists (MORs), which demonstrate improved absorption of EUV light, improved etch resistance, and reduced line edge roughness. Given the significant difference in chemical properties between CAR and MOR, it is imperative for manufacturing viability to understand how existing etch processes are impacted by a switch to MOR in the patterning stack. In this study, we used a high-density plasma reactor to etch a benchmark blanket patterning stack (Fig. 1) containing either a EUV CAR or MOR. We utilized a characterization suite including optical emission spectroscopy (OES), spectroscopic ellipsometry (SE), atomic force microscopy (AFM), and x-ray photoelectron spectroscopy (XPS) to understand the relationships between the plasma conditions (OES), the evolution of the surface chemistry of the resists (XPS), and the resulting etch behavior (SE) and surface topography (AFM). The characteristic plasma processes for a patterned stack etch interact with MOR in a different manner than with CAR, resulting in different physical and chemical impacts to the resist itself, but also via etch byproducts alter the chamber condition and the etch behavior of subsequent etch steps in the patterning stack. We also applied the findings from the blanket stack work on patterned structures to investigate the impacts on pattern fidelity, especially since there is industrial interest to implement MOR for line-space patterning at pitch sizes of 32nm and below. Identifying the underlying mechanisms that lead to differences in the overall stack etch when MOR is used compared to CAR will provide key guidance into the development of process flows that integrate MOR.

10:30am PS-MoM-10 Influence of Nitrogen on Controlling the Etch Selectivity between Tungsten Metal and Dielectric Materials, Indroneil Roy, Pingshan Luan, Jason Marion, Yusuke Yoshida, Peter Biolsi, TEL Technology Center America

Metal gate cut (MGC) last approach has many advantages over traditional poly gate cut such as increased separation length of the gate, improved composite yield, and lower parasitic leakage. In future device structures (e.g. Complementary Field Effect Transistor, CFET), the increase of gate heights along with the decrease of critical dimensions (CD) results in high trench aspect ratios (A.R., calculated by height/CD). This poses a significant etch challenge where the metal etch rate slows down and the etch profile becomes more difficult to control. Simply etching for longer may not be suitable because the hard mask, typically comprised of dielectric materials, will be fully consumed. Thus, a process that provides higher metal-to-dielectric selectivity is highly desirable for MGC applications. In this work, we use blanket films of tungsten (W), which is a typical metal gate material, and SiO_2 and Si_3N_4 , which are typical hard mask materials, to investigate the effect of plasma chemistry in controlling etch selectivity. Particularly, the role of N_2 in a BCl_3/N_2 plasma is explored. We found that the W: SiO_2 and W: Si_3N_4 etch selectivity is governed by the interplay between etchant Cl neutrals, and a passivation effect when N_2 is introduced in the plasma. The

etch amounts of W, Si_3N_4 , and SiO_2 were evaluated using spectroscopic ellipsometry (SE). We found that the W etch amount (E.A.) increases with increasing N_2 flow whereas the dielectric E.A. decreases. However, in a low N_2 flow regime, the Si_3N_4 : SiO_2 selectivity reverses. The behavior of N_2 flow on etch selectivity was evaluated by studying the surface composition of these materials before and after etch using X-ray photoelectron spectroscopy (XPS). The effect of A.R. on etch selectivity was also investigated. The plasma composition is also monitored using optical emission spectroscopy (OES) to understand differences in relative concentration of gas phase species.

10:45am PS-MoM-11 Plasma Etch of Low-K Dielectric (SiOC, SiOCN) at Reduced Temperature, Sang-Jin Chung, University of Maryland, College Park; Pingshan Luan, Adam Pranda, Yusuke Yoshida, TEL Technology Center America; Gottlieb Oehrlein, University of Maryland, College Park

Front-end low-k dielectric (SiOC, SiOCN) are important in complementary field-effect transistors (CFET) for reducing LC delay, minimizing power consumption, and mitigating crosstalk. Future generations of CFETs will also be increasingly stacked to improve device density. Therefore, high-aspect-ratio (HAR) etch and profile defect issues must be considered, as well as the different etch rates of the dielectric/poly-Si materials which will be dependent on the precursor chemistry, substrate temperature, and plasma power.

In this work using an inductively coupled plasma chamber, we will examine SiOC and SiOCN etch both isotropically and anisotropically with varied substrate temperatures (10 °C to -60 °C), precursors, plasma power, and bias voltages. *In situ* ellipsometry will be used to monitor the real time etch behavior, and XPS will be used to probe the composition after etching.

The etch capability of the chamber at cryogenic temperatures has been successfully confirmed.¹ In this work, *in situ* ellipsometry monitoring of the surface can give us a powerful understanding of the chemical properties of the deposited FC film when etching SiOC and SiOCN material, particularly as we lower the substrate temperature when condensation might occur.

In addition to planar etch of these substrates, HAR structures will be used to simulate isotropic etch characteristics of the SiOC and SiOCN materials. A previous study using Si_3N_4 and SiO_2 HAR structures showed significant spontaneous etch when using HFC precursors (CH_2F_2 , CHF_3 , and CF_4/H_2) whereas mostly deposition was seen with FC precursors (C_4F_8).² Our current results show that at low substrate temperature, this isotropic etch was mitigated. The etching characteristic and sidewall profiles of SiOC and SiOCN materials will be compared to SiO_2 and Si_3N_4 .

1. Kihara, Y., Tomura, M., Sakamoto, W., Honda, M. & Kojima, M. Beyond 10 μm depth ultra-high speed etch process with 84% lower carbon footprint for memory channel hole of 3D NAND flash over 400 layers. 2023 IEEE Symposium on VLSI Technology and Circuits, 1–2 (2023).

2. Chung, S.-J., Luan, P., Park, M., Metz, A., & Oehrlein, G. S. Exploring oxide-nitride-oxide scalloping behavior with small gap structure and chemical analysis after fluorocarbon or hydrofluorocarbon plasma processing. J. Vac. Sci. Technol. B 41, 062201 (2023).

11:00am PS-MoM-12 Etch Characteristics of Flexible Low-k SiCOH Thin Films Under Fluorocarbon-Based Plasmas Using Inductively Coupled Plasma-Reactive Ion Etching Process, Rajib Chowdhury, Thomas Poché, SeonHee Jang, University of Louisiana at Lafayette

Flexible electronics have gained considerable attention within the microelectronics industry due to their ability to fold, twist, stretch, and conform to various surfaces. Unlike traditional Si-based electronics, flexible electronic devices are fabricated on substrates such as polymers, metal foils, and flexible glass, enabling applications in displays, wearable devices, and solar cells. However, low-temperature processing is required to produce flexible electronic components due to the low glass transition temperature of polymer substrates. Among various dielectric materials, carbon-doped silicon oxide (SiCOH) is widely used in semiconductor devices for its low dielectric constant (low- k , $k < 4.0$). Photolithography and dry etching are essential for integrating SiCOH into flexible electronics. While the etching properties of SiCOH films on rigid Si wafers are well-studied, research on flexible SiCOH films is limited. In the dry etching process, etchants such as CF_4 , O_2 , and Ar influence the film properties, potentially affecting device performance. This study explores the effect of etching parameters on flexible SiCOH films to advance their application in flexible electronics.

Flexible low-*k* SiCOH films were produced onto flexible indium tin oxide-coated polyethylene naphthalate substrates by plasma-enhanced chemical vapor deposition of a tetrakis (trimethylsilyloxy)silane precursor at ambient temperature. RF plasma powers of 20 and 60 W were utilized for the deposition. An inductively coupled plasma-reactive ion etching process was performed to investigate the etching characteristics of the SiCOH films under CF₄-based plasmas. Each etching gas chemistry was selected among CF₄, CF₄ + O₂, and CF₄ + Ar. The physical, chemical, and electrical properties of the SiCOH films were investigated to determine the effects of etching process parameters on film's properties.

The Fourier transform infrared spectra of the pristine films identified four prominent absorption bands as CH_x stretching, Si-CH₃ bending, Si-O-Si stretching, and Si-(CH₃)_x stretching vibration modes. After etching, the peak area ratio of Si-O-Si stretching mode increased, and that of Si-(CH₃)_x stretching mode decreased. The high-resolution X-ray photoelectron spectroscopy scan found that the peak intensity of the C1s and Si2p peaks decreased after the etching process, and the peak center of the F1s peak shifted depending on etching chemistry. The *k*-values of the pristine SiCOH films deposited at 20 and 60 W were 2.46 and 2.00, respectively. Following etching, the *k*-values of the films at 20 W were reasonably consistent, while those at 60 W increased notably following the etching process.

11:15am PS-MoM-13 Enhancing 24nm Pitch Line / Space by DSA Rectification: A Path to Smoother Lines and Car Extension, Rémi Vallat, Lander Verstraete, Philippe Bézard, Hyo Seon Suh, Laurent Souriau, Kurt Ronse, IMEC, Belgium

The introduction of extreme ultraviolet (EUV) lithography tools has significantly accelerated the scaling of device features, driving advancements in the semiconductor industry. However, as critical dimensions (CD) continue to shrink, the impact of stochastic variability becomes even more pronounced, and the demands for precise patterning grow stricter. Traditional chemically amplified resist (CAR) materials are facing increasing challenges in meeting specifications for roughness, defectivity, and etch budget. As a result, there is a rising interest in exploring alternative resist formulation and patterning schemes, such as directed self-assembly (DSA).

DSA can mitigate the stochastic issue and push EUV patterning close to the resolution limit, thanks to its resilience to small resist variation by extending the use of CAR combined with low dose exposure. For DSA, PS-*b*-PMMA low chi block copolymer is typically used down to 22nm pitch, its phase separation limit. Scaling below 22nm requires the introduction of higher Chi materials to provide a sharper interface with less fluctuations and thus better roughness. [1-3]

In this talk, the case of rectification by DSA at Pitch 24nm Line/ Space CAR patterns (P24 L/S) will be discussed (Figure 1). After introducing the DSA process flow, we will present how to minimize the Line Edge Roughness (LER) and Line Width Roughness (LWR) by optimizing the stack and the dry etch pattern transfer process. Finally, we will discuss how to further improve the roughness (both LER and LWR) at BCP level (for both PS-*b*-PMMA and HighChi) and how the improved roughness is modulated during the pattern transfer down to the layer below.

[1] M. P. Stoykovich, K. C. Daoulas, M. Muller, H. Kang, J. J. de Pablo, P. F. Nealey, *Macromolecules*, 2010, 43, (5), 2334-2342.

[2] R. Ruiz, H. Kang, F. A. Detcheverry, E. Dobisz, D. S. Kercher, T. R. Albrecht, J. J. de Pablo, P. F. Nealey, *Science*, 2008, 321, (5891), 936-939.

[3] G. Singh, N. Nair, F. Gstrein, R. Schenker, Proc. SPIE PC12953, Optical and EUV nanolithography XXXVII, PC129530G, 2024.

11:30am PS-MoM-14 Dry Etch Challenges Towards the High NA EUV Lithography Patterning Era, Sara Paolillo, Stefan Decoster, Philippe Bezaud, Remi Vallat, Vincent Renaud, Viktor Kampitakis, Annaelle Demaude, Laurent Souriau, Bhavishya Chowrira, Dieter Van Den Heuvel, Victor M. Blanco Carballo, Syamashree Roy, Shubhankar Das, Frederic Lazzarino, IMEC, Belgium

INVITED

Pitch scaling remains a fundamental driver of semiconductor technology advancement. To print smaller feature sizes, Extreme Ultraviolet (EUV) lithography was introduced starting from the 7 nm technology node. As we are approaching the scaling limits of Low Numerical Aperture (NA) EUV, High NA EUV lithography is being proposed as a solution for patterning sub-24 nm pitch structures in a single exposure step. Additionally, multi-patterning schemes are being employed to extend capabilities beyond the lithographic resolution limit. In all cases, to support this ongoing

miniaturization, it is crucial to ensure the precise transfer of the photoresist patterns into underlying layers through plasma etching.

This presentation will highlight the opportunities and evolving challenges associated with dry etching at current resolutions and as we transition into the high NA era. As device features shrink, the lithography trade-off between resolution, sensitivity, and features roughness becomes increasingly challenging to balance; improving one comes at the expenses of the others. One major challenge is addressing local photoresist non-uniformities, such as height variations and residues after development, which can lead to defects (breaks or bridges) after pattern transfer. These effects become even more pronounced when using thinner resists required for High NA lithography. To mitigate these issues and improve pattern fidelity, strategies such as descum processes, enhanced etch selectivity, and novel break-healing techniques are being explored. Another key challenge related to the aforementioned trade-off is minimizing features roughness, including line roughness (LER/LWR), local CD non-uniformity (LCDU), and tip-to-tip (T2T) variability. We will discuss several methods to mitigate these effects, like directional etching, directed self-assembly, and precise tuning of etching parameters.

Beyond the challenges related to working at the limits of the lithographic process, patterning increasingly smaller features in the final stack also presents significant challenges. Achieving high selectivity, vertical profiles, minimal mask erosion, low feature roughness, and preventing line bending demands continuous advancement in both etching techniques and patterning stack design. We will discuss how the choice of materials within the stack and their properties affect patterning performance.

Finally, we will present case studies from IMEC where high NA exposure has been introduced, and we will discuss the results of pattern transfer.

12:00pm PS-MoM-16 Next Generation Interconnect Patterning of Ruthenium Using Advanced RIE Processing, Shravana Kumar Katakam, Harsimran Singh, IBM Research Division, Albany, NY; Nicholas Joy, Dayun Yan, TEL Technology Center, America, LLC, Albany, NY; Christopher Penny, Koichi Motoyama, Hosadurga Shobha, Yann Mignot, Joe Lee, IBM Research Division, Albany, NY

The demand to scale interconnects to tighter pitch is increasing substantially to match the logic scaling trend and new interconnect materials like Ruthenium (Ru) are being considered as a replacement of Copper (Cu) interconnects for 18nm Pitch (P18) and beyond. There are unique patterning challenges associated with Ru interconnects using subtractive Reactive Ion Etching (RIE). It has been reported in our previous work that Hard Mask (HM) swelling during RIE is one of the major challenges at these tighter pitches and demands an innovative RIE solution. It was demonstrated that P18 patterning was successful by implementing a multi-step etch process. However, the process margin further shrinks when the pitch is reduced to P16 and beyond and needs further improvements in the RIE process. The challenges for P16 are similar to P18 and the main issue continues to be hardmask swelling from HM re-deposition during the etching process especially for high aspect ratio lines. Furthermore, in addition to HM swelling due to tighter space CDs, Line Edge Roughness (LER) and Line Width Roughness (LWR) are additional contributing factors impacting the yield at these pitches. To this end, the RIE solution not only demands a recipe without hardmask swelling but also with reduced roughness. As a continuation of our previous work, we present here a unique RIE solution which not only has very minimal hardmask swelling but also has a significant reduction in LER compared to multi-step process. A special passivation process has been implemented for reducing the roughness in conjunction with advanced pulsing capabilities which resulted in successful patterning of Ru interconnect at P16. The effects of changes to the pulsing conditions and passivation gas addition are evaluated, and the results are discussed.

Plasma Science and Technology Room 201 ABCD W - Session PS-MoA

Plasma Modelling Focused on CCP

Moderators: Mingmei Wang, Lam Research Corporation, Du Zhang, TEL Technology Center America

1:30pm PS-MoA-1 Plasma Prize 2024 Award Talk: Some Tales from Our Model Validation Adventures, Shahid Rauf, Han Luo, Peng Tian, Jun-Chieh Wang, Xingyi Shi, Tianhong Wang, Nakul Nuwal, Rupali Sahu, Kallol Bera, Jason Kenney, Applied Materials, Inc.; Manuel Schroeder, Jan Guttman, Niklas Friedrichs, Ihor Korolov, Julian Schulze, Ruhr-University Bochum, Germany

INVITED

Plasma modeling has emerged as a crucial tool in the design of plasma equipment within the semiconductor industry. These models are also extensively utilized for exploratory studies and to enhance the understanding of the physics underlying major plasma processing applications. Given the inherent complexity of plasmas, model validation – through testing against experimental measurements – is essential for developing credible models. We report on a multi-year, broad collaboration between our modeling and experimental teams aimed at systematically examining models for capacitively and inductively coupled plasmas. The modeling efforts have employed particle-in-cell (PIC), fluid, and hybrid models, allowing for a comparative analysis of the modeling techniques. Experimental diagnostics have included Langmuir probes, phase-resolved optical emission spectroscopy (PROES), and retarding field ion energy analyzers (RFEA). Ion energy distribution function (IEDF) measurements using the RFEA in argon (Ar) and oxygen (O₂) plasmas highlight the significance of kinetic effects in low-pressure capacitively coupled plasmas (CCP). In low-density CCPs, where the sheath thickness ranges from 5 to 10 mm, collisions distort the IEDF even at pressures as low as 2.5 Pa. Experimentally validated two-dimensional (2D) models demonstrate the impact of plasma non-uniformity on the symmetry of the ion angular distribution function (IADF). PROES and modeling have been employed to investigate multi-frequency CCPs with tailored voltage waveforms for the low-frequency (LF) component. One key finding of this combined modeling-experimental study is the role of the LF in power coupling by the high-frequency (HF) source, which affects both the spatial and temporal characteristics of plasma production. To test 2D plasma models, we have examined radio frequency (RF) hollow cathode discharges (HCD) in Ar and O₂ with several HCD geometries, RF frequencies, and pressures ranging from 5 to 100 Pa. Kinetic effects are significant across the entire pressure range, underscoring the importance of employing kinetic or hybrid models in simulating CCPs. Our collaborative efforts are now focused on inductively coupled plasmas (ICP), with ongoing tests of models for pulsed ICPs that transition between the E, E/H, and H modes of operation. Preliminary results from ICP diagnostic and modeling work are presented.

2:00pm PS-MoA-3 Experimental Validation of a Stability Model for Capacitively Coupled Plasmas, Omar Alsaed, North Carolina State University; Brian Bentz, Sandia National Laboratories; Benjamin Yee, Brett Scheiner, Chenhui Qu, Meenakshi Mamunuru, Lam Research Corporation; Amanda Lietz, North Carolina State University

Self-organization in radiofrequency plasmas is a commonly occurring phenomenon that can be detrimental to wafer-scale uniformity in semiconductor manufacturing. Recent theoretical work has proposed that thermoelectric electron energy transport within a fluid electron framework drives the onset and growth of these instabilities. A theoretical stability criterion was previously tested experimentally in inductively coupled plasmas and numerically in capacitively coupled plasmas, showing good agreement. However, the underlying driving mechanism remains unverified, with conflicting reports in the literature, and experimental validation efforts have been lacking in capacitively coupled plasmas. This work presents experimental measurements of the unstable mode wave number and growth rates in symmetric planar capacitively coupled plasmas at moderate pressures (0.1–10 Torr) in electropositive chemistries (Ar, He, N₂). Measurements were performed using laser collision-induced fluorescence (LCIF), optical emission spectroscopy (OES), and high-speed imaging. The experimental results are compared against theoretical predictions of stability as a function of gap size, gas pressure, and chemical composition, providing insights into the nature of instability in moderate-pressure radiofrequency plasma systems.

* Work funded by Lam Research Corporation, U.S. DOE Early Career Research Program Award DE-SC0025621, and by Sandia National Laboratories' Plasma Research Facility, funded by the U.S. Department of Energy Office of Fusion Energy Sciences. Sandia is managed and operated by NTESS under DOE NNSA contract DE-NA0003525.

2:15pm PS-MoA-4 Radio-Frequency Power Pulsing of Intermediate Pressure Electronegative Capacitively Coupled Plasma, Rupali Sahu, Kallol Bera, Shahid Rauf, Applied Materials Inc.

Plasma processing of wafers utilizes ion and radical based surface processes for high throughput, low-temperature operation. In cases where radical-surface reactions are preferred over ions, power pulsing can be utilized to control the radical-to-ion ratio. In the most basic form, RF pulsing involves switching CCP power between ON and OFF states at a certain pulse frequency. Radicals and ions are generated during the pulse ON phase. During the pulse OFF phase, ions and electrons disappear quickly while the radicals are lost more gradually. Hence, the longer the OFF time is, the higher is the cycle-averaged radical-to-ion flux ratio. A larger pulse OFF time can lead to difficulties in plasma reignition when the power is turned ON, as very few electrons are left for bulk avalanche ionization. These factors determine the stable operating regime for RF pulsing in an electropositive plasma. Plasma modeling shows stable RF pulsing operation is usually achieved at high pulsing frequency and large duty cycle, which is consistent with experimental data.

Strongly electronegative plasmas are characterized by negative ion density being much higher than that of electrons. In this study, we use plasma modeling to analyze pulsing behavior of electronegative capacitively coupled plasmas. This study has been done for He-NF₃ gas mixture at intermediate pressure (~a few Torr). The pulsing frequency and duty-cycle are varied. Modeling results show that strongly electronegative plasma exhibits some counter-intuitive results, such as stable pulsing operation being limited to low pulsing frequency and delayed ignition during pulse ON for certain process conditions. Mechanisms of power absorption in the plasma and electron generation are examined for all cases. It was seen that Penning ionization of Helium sustains electron density during the pulse OFF period, until eventually running out of the excited states of Helium. During the pulse ON phase, the power is preferentially absorbed by the charged specie with highest conductivity, which turns out to be F⁻ ions in some cases, causing a delay in plasma re-ignition. If the delay is larger than the pulse ON duration, the plasma fails to reignite, and the plasma can't be sustained. These mechanisms allow for a pulsing window which exhibits large pulse ON times and can afford to have relatively longer pulse OFF times compared to plasmas that don't have penning ionization reactions.

2:30pm PS-MoA-5 Hybrid Kinetic-Fluid Methods of Plasma Modeling, Vladimir Kolobov, University of Alabama at Huntsville

INVITED

Low-temperature plasma (LTP) is a quasi-neutral mixture of electrons, ions, and neutral species. Its highly non-equilibrium state is beneficial for numerous technological applications. Several methods of particle transport, ranging from fully kinetic to hydrodynamic, have been developed to model LTP systems. In our presentation, we will highlight some important recent advances in the field and discuss the challenges of selecting appropriate models for efficient and accurate simulations.

The disparity of scales and non-local electron kinetics are the most challenging aspects of plasma simulations [1]. The attached Figure shows the key steps in selecting suitable models for electrons in non-magnetized LTP depending on the characteristic temporal and spatial scales. Solutions of the complete Boltzmann kinetic equation by particle-based (PIC) and phase-space-grid methods remain expensive for practical engineering. The disparity of momentum and energy relaxation scales for electrons allows for reducing the phase space dimensionality using angular moments. The dominance of elastic scattering for slow electrons results in a quasi-diffusion equation for the Electron Energy Distribution Function (EEDF) in coordinate-energyspace. Small-angle scattering and continuum energy loss are suitable for fast runaway electrons. Fluid models become adequate when the spatial/temporal scales exceed the electron energy relaxation length/time. Depending on the Coulomb collision frequency, two fluid model varieties could be justified (see attached Figure description). Selecting appropriate models for electrons capturing non-local kinetic effects is poorly understood and will be the focus of our presentation.

Our presentation will show examples of hybrid kinetic-fluid simulations of AC and DC plasma sources, illustrating the importance of non-local kinetic effects. We will explain why maintaining plasma in the dynamic and stratified regimes is the most energy efficient [2]. Finally, we will review recent efforts to use Physics Informed Neural Networks (PINNs) for plasma

simulations [3] and discuss perspectives for integrating machine learning algorithms for solving inverse problems in plasma science and engineering.

[1] V.I. Kolobov and R.R. Arslanbekov, Towards adaptive kinetic-fluid simulations of weakly ionized plasmas, *J. Comput. Phys.* 231 (2012) 839

[2] V. I. Kolobov and Y. B. Golubovskii, The principle of minimal power, *Plasma Sources Sci. Technol.* 31 (2022) 094003

[3] V. Kolobov and L. Schoenbaum, Development of grid-based and PINN solvers for electron kinetics in collisional non-thermal plasmas, <https://arxiv.org/abs/2412.16706>

3:00pm PS-MoA-7 Regression-based Circuit Estimation of Collisional Sheath in Moderate Pressure Capacitively Coupled Plasma, Sathya Ganta, Abhishek Verma, Kallol Bera, Applied Materials, Inc.

Capacitively coupled plasma chambers are widely used in the semiconductor industry for various deposition and etching applications. Many of these applications require moderate pressure (a few Torr) plasma that provides the required electron density, ion flux and sheath potential/ion energy. At these pressures, the sheath is collisional and cannot be studied by most of the existing analytical techniques that are suitable for collision-less sheaths. In this paper, we begin with the standard circuit model representing a sheath with capacitive element capturing sheath voltage to sheath displacement current relation, resistive element depicting sheath voltage to sheath ion current relation and diode element corresponding to sheath voltage to sheath electron current relation. We then added features to the sheath circuit model that captured the collisional aspects of the sheath at moderate pressure like delay in the electron and ion current responses to sheath voltage and separate resistive elements to capture ion currents at different harmonics. The parameters that define the new collisional sheath circuit model were estimated for any given set of plasma process conditions by comparing sheath voltage and electron, ion and displacement current data obtained from corresponding 1D fluid Argon gas capacitively coupled plasma (CCP) simulations. Regression-based statistics were then used to build a relationship between plasma process conditions like pressure / RF voltage / RF phase and the estimated parameters of the new collisional sheath circuit model. We then used this regression relationship, and an electromagnetic model implemented with the new collisional sheath circuit to predict electron, ion, displacement currents and sheath voltage (i.e., on-wafer ion fluxes and ion energies) for Argon gas capacitively coupled plasma over a wide process span without running the computationally intensive plasma simulations.

3:15pm PS-MoA-8 The Impact of Tailored Voltage Waveforms on Reaction Rates in Capacitively Coupled Plasma Ar/O₂ Discharges, Syed M. Zulqarnain, Amanda M. Lietz, North Carolina State University; James Prager, Timothy Ziemba, EHT Semi

Capacitively coupled plasmas (CCPs) are extensively employed in the selective and anisotropic etching of semiconductor materials, as well as in the deposition of thin films. Tailored voltage waveforms (TVW) offer a promising approach to manipulating plasma dynamics and controlling ion and electron energy distributions. These TVW may also provide reduced differential charging within high aspect ratio features, which can cause problematic ion deflection. In many industrial applications, RF CCPs operate with diverse mixtures of electronegative and electropositive feed gases, resulting in different species of positive and negative ions, neutral radicals, and electrons in the plasma volume. Mixtures containing oxygen (O₂) are vital for many etching processes. O₂ significantly contributes to the removal of polymer films during etching by facilitating their oxidation, which helps prevent the loss of ions in the polymer and accelerates the etching of the target material. The control of ion energy and flux is critical for optimizing these processes and offers significant advantages for controlling plasma chemistry during processing. This computational investigation delves into the plasma dynamics of argon/oxygen admixtures in a CCP biased by a triangular-shaped TVW, its impact on ionization rates, and the transport mechanisms of ions and electrons. In this study, a dual-frequency capacitively coupled argon/oxygen discharge at 5 mTorr pressure with a high-frequency (60 MHz) sinusoidal voltage waveform applied to the upper electrode and a low-frequency (400 kHz) triangular-shaped TVW bias applied to the lower electrode is simulated, using Monte-Carlo collision-based, particle-in-cell code (EDIPIC). As the O₂ concentration increases, the electronegativity of the discharge is expected to influence the distribution of positive ions and electrons and the ionization dynamics of the discharge. We explored the impact of TVW on discharge behavior, with particular emphasis on the evolution of ionization rates and electron dynamics within a single low-frequency waveform cycle. The analysis focused on the electric field reversals that may occur during the positive phase of the waveform,

examining their effects on ionization rates and power deposition as O₂ concentration varies. Additionally, the study compared the variations in ionization and excitation levels in the bulk plasma between TVW and conventional sinusoidal waveforms. This study elucidates the interplay between plasma electronegativity, reactive chemistry, and tailored voltage waveforms in manipulating plasma dynamics to inform the optimization of semiconductor etching and deposition techniques.

3:30pm PS-MoA-9 Intermediate Pressure Capacitively Coupled Plasma Model Validation, Kallol Bera, Rupali Sahu, Nakul Nuwal, Shahid Rauf, Applied Materials, Inc.

Radio-frequency (RF) capacitively coupled plasmas (CCP) at intermediate pressure (~ a few Torr) are widely used in advanced plasma processing in the semiconductor industry. However, the plasma behavior in this pressure regime is not well characterized. Plasma modeling, validated with experimental data at these pressures, enhances the understanding of plasma behavior that is crucial for the plasma chamber design and process development. CCPs with Argon, an electropositive gas, and Oxygen, an electronegative gas, are simulated using a one-dimensional fluid-MCS hybrid plasma model at intermediate pressures and compared with experimental data. Key parameters such as electrode voltage, current, and phase, as well as electron densities, are compared and analyzed. Additionally O density measurements are compared to simulation results for Oxygen plasma. Our model includes continuity equations for charged and neutral species, drift-diffusion approximation for electron flux, momentum conservation equation for ions, energy conservation for electrons, and the Poisson equation for electric potential. The secondary electrons emitted from the surface are treated kinetically using a Monte Carlo model as they accelerate across the sheath. This kinetic secondary electron model is coupled to the fluid bulk plasma model to capture the contributions from these secondary electrons to species production and electron power deposition. A Particle-in-cell model Monte Carlo Collision (PIC-MCC) model is employed to understand the kinetic behavior, and to compare with the hybrid model. Kinetic effects are found to be significant at intermediate pressure highlighting the importance of incorporating kinetic effect at these pressures. The Druyvesteyn Electron Energy Distribution Function (EEDF) is found more appropriate than Maxwellian distribution, consistent with PIC-MCC model results. For Ar plasma, dimers are found to play a crucial role at these pressures. The surface sticking coefficient of atomic oxygen is a critical factor in determining plasma density and O density for Oxygen plasma.

4:00pm PS-MoA-11 Modeling insights into amorphous carbon etching by SO₂/O₂ low-pressure plasma, Dmitry Levko, Mingmei Wang, Lam Research Corporation

In this work, we will explore the influence of the oxygen partial pressure on the amorphous carbon film etching by low-pressure SO₂/O₂ plasma for the experimental conditions of Ishikawa *et al.*, *Applied Surface Science* **645**, 158876 (2024). We will use zero and one-dimensional plasma models to develop and validate both gas-phase and surface reactions mechanisms of SO₂/O₂ plasma in contact with carbon mask. We will discuss the influence of oxygen partial pressure on the plasma parameters such as the main ion and reactive species densities, plasma potential and peak-to-peak voltage. We will also discuss the influence of oxygen on the amorphous carbon etch rate.

4:15pm PS-MoA-12 Pulsed Power Strategies for Plasma Etching of High Aspect Ratio Features Using Fluorocarbon Gas Mixture for Feature Charging Control, Yifan Gui, Yeon Geun Yook, Chenyao Huang, Mark J. Kushner, University of Michigan

In microfabrication, plasma etching of high aspect ratio (HAR) features with precision remains a significant challenge largely due to feature charging effects that can lead to profile distortion and etch stop. Feature charging occurs when there is an imbalance in the flux of ions and electrons to inside surfaces of features, leading to the creation of local electric fields that deflect incoming charged species and distort ion trajectories. Previous experimental and modeling studies have shown the sensitivity of feature charging on energy and angular distributions (EADs) of charged species incident onto the wafer. Potential remedies for feature charging include pulsed plasma operation, tailored bias waveforms, and the introduction of electronegative gases to suppress electron density or promote charge neutralization. These strategies aim to balance ion and electron fluxes or temporarily neutralize accumulated charge to mitigate defects within the HAR features. For example, the use of pulsed power is believed to produce a cycle of charging and discharging of the feature as the fluxes and EADs of the charged particles are modulated.

In this work, we discuss a computational investigation of pulse power strategies for controlling the fluxes of charged particles to wafers in capacitively coupled plasmas (CCPs) with the goal of mitigating feature charging. The Hybrid Plasma Equipment Model (HPEM), a modular simulator designed to address the behavior of low-pressure plasma systems, was used to investigate the evolution of incident fluxes and EADs of charged particles during pulse-on and -off periods in multi-frequency CCPs using fluorocarbon gas mixtures and mixtures amenable to cryogenic etching. The consequences of utilizing different modes of pulse operation (low frequency, high frequency, dc) and gas mixtures on the EADs and charged species flux will be discussed in relation to minimizing feature charging.

Work supported by Lam Research, Samsung Electronics and Department of Energy Office of Fusion Energy Sciences.

4:30pm PS-MoA-13 Application of a Structured Showerhead Electrode in Plasma Enhanced Chemical Vapour Deposition: Modeling and Experimental Study, *Montu Bhuvu, Geoff Hassall, James Ellis, Gregory Daly*, Oxford Instruments Plasma Technology, UK; *Erik Wagenaars, James Dedrick*, University of York, UK

The application of a structured showerhead electrode in improving the film thickness uniformity is investigated for large diameter substrate processing using plasma enhanced chemical vapor deposition (PECVD). The fluid-kinetic simulations are carried out using the hybrid plasma equipment model (HPEM) accessed via the Quantemol Virtual Tool (QVT) interface. The modelling results are experimentally verified with the optical actinometry in Ar/O₂ plasma to capture the structured electrode effects on the radial atomic oxygen concentration. The above investigations are conducted at an intermediate pressure regime of 1-2.5 Torr in the Oxford Instruments PlasmaPro-100 PECVD test reactor. The simulation model is further applied to investigate the performance of the multi-cavity structured showerhead in SiH₄ chemistry accessed via the Quantemol Database (QDB). The simulation model indicates that the showerhead cavity rings play a vital role in controlling the radial plasma profile adjacent to the substrate. Finally, the designed structured showerheads, based on the simulations, are tested in the clean room environment for SiO₂ deposition, and the improvements in the process results are characterised against the conventional planar showerhead.

4:45pm PS-MoA-14 Kinetic and Hybrid Modeling of a Radio Frequency Hollow Cathode Discharge and Comparison with Experiments, *Nakul Nuwal, Kallol Bera, Han Luo, Xingyi Shi*, Applied Materials Inc.; *Shahid Rauf*, Applied Materials, USA; *Jan Guttman*, Applied Materials Inc.; *Ihor Korolov, Julian Shulze*, Ruhr Universität Bochum, Germany

Radio frequency (RF) hollow cathode discharges (HCD) are used in various semiconductor manufacturing processes such as material etching and deposition. HCD cathodes have cavities, and the plasma forms inside these cavities under the right conditions. In the HCD, RF sheath heating as well as secondary electron acceleration can lead to plasma production. In this work, plasma simulation results for argon and oxygen HCDs are compared with plasma diagnostics measurements using non-invasive methods. These measurements include the emission spectra of plasma discharge using Phase Resolved Optical Emission Spectroscopy (PROES), which provides the spatio-temporal excitation rate of important species in the discharge. We use both kinetic and hybrid plasma models in this work to understand the plasma dynamics and elucidate with the experimental observations. The Particle-In-Cell with Monte Carlo Collisions (PIC-MCC) model includes evolution of charged particles and electrostatic field along with charged particle collisions with the neutral species using a Monte Carlo approach. The hybrid model only treats the electrons as particles and includes a fluid model for the other charged species. In both models, the charged species' densities are coupled with the Poisson's equation to calculate the electric potential, enabling a self-consistent plasma simulation. Plasma simulations are performed for different pressures, voltages, and feed gases (Ar & O₂). Our simulation results show good agreement with the spatio-temporal experimental measurements of metastable argon excited state at low pressures. With increase in voltage, the excited species is found to penetrate further into the hollow cathode slot. The modeling results also indicate that the secondary electron emission coefficient from surfaces significantly influences the plasma behavior.

5:00pm PS-MoA-15 Molecular Dynamics Analysis of Transport Properties and Gap-Filling Mechanisms in Flowable Chemical Vapor Deposition Using TEOS-Based Plasma, *Hu Li*, Tokyo Electron America Inc.; *Takeo Nakano, Masaaki Matsukuma*, Tokyo Electron Technology Solutions Ltd., Japan; *Jianping Zhao, Peter Ventzek*, Tokyo Electron America Inc.,

Flowable chemical vapor deposition (Flowable CVD) is an advanced deposition technique widely used for effectively filling nanoscale structures with complex geometries using plasma-generated oligomers. This method is particularly crucial in semiconductor manufacturing and advanced memory device fabrication, especially for structures with high aspect ratios at dimensions of tens of nanometers. Conventional CVD approaches often encounter incomplete gap-filling, resulting in void formation that significantly compromises device performance. Flowable CVD addresses these challenges by employing oligomers synthesized through plasma-induced polymerization reactions, which exhibit fluid-like properties at relatively low deposition temperatures. This inherent fluidity enables superior gap-filling without the need for additional oxidation steps.

However, despite these evident advantages, the fundamental mechanisms underlying oligomer formation, fluid dynamics, and transport processes in Flowable CVD remain inadequately understood, resulting in occasional practical difficulties such as void formation. Therefore, gaining comprehensive insights into oligomer transport phenomena and deposition mechanisms is essential for optimizing Flowable CVD processes to achieve reliable, void-free film deposition.

This study addresses these critical gaps in understanding by examining the transport properties of oligomer species likely formed in tetraethoxysilane (TEOS)-based plasma environments. Molecular dynamics (MD) simulations were performed to quantitatively evaluate essential characteristics of oligomer liquids, including surface tension, viscosity, and contact angle interactions on wafer surfaces. These analyses provide valuable insights into how such physical properties affect overall gap-filling performance.

Our simulation results indicate that the viscosity of TEOS-derived oligomers increases with decreasing temperature. Furthermore, oligomers with higher molecular weights exhibited relatively lower viscosities, suggesting longer durations required for complete gap-filling. This research establishes clear correlations between oligomer transport properties and deposition efficiency, thereby contributing valuable knowledge to the fundamental mechanisms governing gap-filling and addressing related process challenges.

5:15pm PS-MoA-16 Validation of Fluorocarbon Containing Gas-Phase Reaction Mechanisms for Computational Modeling of Commercial Atomic Scale Processing Plasmas, *Jordyn Polito, Ben Harris, Geoff Hassall, James Ellis*, Oxford Instruments Plasma Technology, UK

Semiconductor hardware companies require in-depth understanding of plasma systems to address customer support challenges and to inform hardware and process design. Robust, reliable plasma models can be used to increase understanding of plasma systems and aid in rapid solutions to industrial challenges. However, industrial-scale plasma modeling is often challenged by a lack of in-situ diagnostics to validate process-relevant plasma chemistry reaction mechanisms. In this work, the impacts of utilizing reliable plasma modeling techniques together with validated process-relevant reaction mechanisms will be considered from an industrial perspective. Here, a reaction mechanism for CF₄/O₂ is compiled and used in a 0D global plasma model to predict reactive neutral and ion densities and electron temperatures in the plasma region of an inductively coupled plasma used for reactive ion etch processes (RIE). Experimental measurements obtained in an Oxford Instruments Plasma Technology Cobra 300 reactor will be used to validate the reaction mechanism and predict operating conditions that lead to optimal process outcomes.

Electronic Materials and Photonics

Room 207 A W - Session EM1+AP+CPS+MS+PS+QS+SM+TF-TuM

Emerging Frontiers in Quantum Materials and Devices

Moderator: Mollie Schwartz, MIT Lincoln Laboratory

8:30am **EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-3 Rapid, Atomic-Scale Smoothing of GaSb(111)A Surfaces During Molecular Beam Epitaxy, James Rushing, Paul Simmonds, Tufts University**

InAs/Ga(In)Sb quantum wells (QWs) with a broken gap band alignment can behave as a quantum spin hall insulator (QSHI) with an insulating bulk and topologically protected helical edge states [1-2]. QSHIs could be a key component in spintronic and topological quantum computing applications [2-3]. Producing a topological phase transition in InAs/Ga(In)Sb QWs requires precise control of QW thickness, composition and quality, particularly at the heterointerfaces. Additionally, our calculations suggest QWs grown on (111) surfaces could provide benefits over (001) due to the higher symmetry and out-of-plane polarization effects of this surface.

While exploring the MBE growth of InAs/Ga(In)Sb QW heterostructures on GaSb(111)A, we discovered an exciting and confounding phenomenon that seems to be unique to crystal growth on III-Sb(111)A surfaces. Ga(In)Sb(111)A frequently exhibits an extremely rough morphology characterized by pyramidal peaks covering the entire surface. We show that rough III-Sb surfaces (pyramidal features >70nm in height; rms roughness >10nm), can be smoothed to atomically flat surfaces (<3nm height features; <0.5nm rms roughness) in a matter of seconds by exposing them to an arsenic over-pressure. We first observed this phenomenon when rough GaInSb(111)A surfaces became atomically flat after capping with just 8nm of InAs. After reducing the thickness of this InAs layer to a single monolayer and still observing the same surface smoothing effect, we found that we could achieve almost identical results by simply exposing the rough GaSb(111)A to an arsenic flux. These results suggest that arsenic is the primary mover in these profound morphological changes. Our recent results show that the smoothing can be accomplished with As₄ or As₂, and with a wide range of arsenic beam equivalent pressures, from 5x10⁻⁷ to 1x10⁻⁵ Torr.

We will describe our efforts to gain control and understanding of this phenomenon through the modulation of arsenic exposure time, flux, and terminating III-Sb material. This powerful new MBE technique will allow us to reliably achieve smooth heterointerfaces in (111)-oriented InAs/Ga(In)Sb QWs for novel, high-quality QSHIs. More broadly, we believe that this approach will enable the growth of a wide array of III-Sb-based nanostructures on (111)A surfaces for other electronic and photonic applications.

1. Krishtopenko and Teppe. *Science Advances* **4**, eaap7529 (2018)
2. Avogadri et al. *Physical Review Research* **4**, L042042 (2022)
3. Du et al. *Physical Review Letters* **119**, 056803 (2017)

8:45am **EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4 Benchmarking Different NbTiN Sputtering Methods for 300 nm CMOS-compatible Superconducting Digital Circuits, Adham Elshaer¹, Jean-Philippe Soulié, Daniel Perez Lozano, Gilles Delie, Ankit Pokhrel, Benjamin Huet, IMEC Belgium; Margriet J. Van Bael, KU Leuven and Imec, Belgium; Daan Buseyne, KU Leuven, Belgium; Blake Hodges, Seifallah Ibrahim, Sabine O'Neal, Imec USA; Zsolt Tökei, Imec Belgium; Anna Herr, Quentin Herr, Imec USA**

The NbTiN films presented here are CMOS-compatible and were developed for metallization purposes in superconducting digital circuits [1-5]. Those circuits use NbTiN for Josephson junctions and capacitors electrodes, as well as for wiring. Superconducting digital circuits initially relied on Nb in the early days. NbTiN is a better candidate/replacement due to its higher thermal budget and better chemical stability [1-5]. In this study, the properties of superconducting NbTiN thin films deposited using two different sputtering methods have been compared. One method used multiple targets (MT) co-sputtering (Nb and Ti targets), while the other used a NbTi single target (ST). Benchmarking metrics used for comparison include: superconducting, electrical, as well as morphological properties. All films show a high T_c, ranging from 13.3 K to 15.1 K. Compared to MT, ST NbTiN films showed consistently lower resistivity and better sheet

resistance (Rs) wafer-level uniformity (49 points wafer-map). For instance, 50 nm MT film had a Rs relative standard deviation (Stddev%) of 15.5%, while for the ST NbTiN films, Rs Stddev% showed a 2-fold improvement at 7.8%. Upon annealing of the ST NbTiN films at 650°C, the Rs uniformity further improved, reflected by a lower Stddev% at 4.5%. AFM data show similar results for MT and ST films, ~1.07 nm and 1.09 in the center and 0.73 nm and 0.71 nm at the edge of the wafers, respectively. Furthermore, XRD theta-2theta scans have been performed showing the 200 and 111 peaks for NbTiN orientations. Results show that the MT and ST films have different/signature 200/111 peak intensity ratios for the as deposited films. ST NbTiN films have a lower 200/111 peak ratio. However, after annealing at 650°C, the ST films 200/111 peak ratio increases, and surpasses that of the MT NbTiN films. This change suggests a change in the ST film disorder and grain size after annealing. The impact of the ST NbTiN film thickness on properties has also been studied. The T_c shows an increase as a function of thickness, from 9.6 K for 7 nm, to 14.3 K for 50 nm, up to 14.9 K for 200 nm films. Certainly, the ability to tune the superconducting properties of NbTiN, makes them appealing from a stack engineering perspective. Both MT and ST NbTiN properties can be tailored using deposition conditions such as: power, partial pressure and post deposition annealing [3]. However, MT NbTiN films 300 mm wafer-level Rs non-uniformity represents a limiting factor for scaling superconducting devices. Annealed NbTiN ST films on the other hand, show a 3.4-fold Rs wafer uniformity improvement while maintaining properties tunability.

9:00am **EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-5 Controlling the Properties of Epitaxially Grown Topological Semimetals, Kirstin Alberi, National Renewable Energy Laboratory**

INVITED

Three dimensional topological semimetals (TSMs) exhibit a wide range of interesting properties, including high carrier mobility, large magnetoresistance, anomalous transport behavior, broadband optical absorption and non-linear optical responses. Epitaxial thin film synthesis offers a practical platform for manipulating composition, defects and disorder in these materials, offering a window into approaches for manipulating their properties. In this talk, I will discuss insights into the relationships between structure and composition and the resulting properties revealed through careful control of growth conditions. Focused examples include the impact of point defects and impurities on electron transport in the Dirac TSM Cd₃As₂ and the formation and behavior of domain boundaries in the Weyl TSM TaAs.

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9:30am **EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-7 Photon Down-Conversion of Yb-Doped CsPb(Cl_{1-x}Br_x)₃ to Low-bandgap Metal Halide Perovskites, Yutong Ren², Princeton University; Igal Levine, The Hebrew University of Jerusalem, Israel; Dan Oron, David Cahen, Weizmann Institute of Science, Israel; Antoine Kahn, Princeton University**

Quantum cutting represents a transformative strategy to mitigate thermalization losses that typically occur when high-energy photons are absorbed by semiconductors.^{1,2} Recent advances have extended this concept from rare-earth doped crystals to semiconductor-rare-earth hybrid systems, particularly those utilizing halide perovskite absorbers, thereby exploiting their exceptional optoelectronic properties.

In this study, we focus on Ytterbium (Yb) -doped CsPb(Cl_{1-x}Br_x)₃, a metal halide perovskite that absorbs visible light and exhibits intense near-infrared (NIR) photoluminescence—a clear signature of efficient quantum cutting. Upon excitation with visible light, the doped perovskite converts the absorbed energy into two NIR photons, with the emission energy closely matching the optimized bandgap of a Sn-Pb based perovskite absorber. This spectral alignment is critical for enabling effective energy transfer between the quantum cutting layer and the absorber.

Our investigation focuses on elucidating the structural and electronic properties of the interfaces between Yb-doped $\text{CsPb}(\text{Cl}_{1-x}\text{Br}_x)_3$ and Sn-Pb based perovskite films. By employing a suite of advanced spectroscopic techniques—including ultraviolet photoelectron spectroscopy, inverse photoemission spectroscopy, time-resolved photoluminescence (tr-PL), and time-resolved surface photovoltage (tr-SPV)—we systematically examine how the quantum cutting layer, the absorber layer, and their interfacial region collectively influence energy transfer efficiency. In particular, the complementary tr-PL and tr-SPV analyses unambiguously determine the dominant interfacial charge transfer and recombination processes, and thus gain control over the interfacial charge transfer. By integrating Yb-doped $\text{CsPb}(\text{Cl}_{1-x}\text{Br}_x)_3$ with customized Sn-Pb perovskite absorbers, our approach shows promise for pushing the boundaries of conventional efficiency limits while also offering a cost-effective strategy for enhanced energy conversion.

1. Wegh, R. T. et al. Quantum cutting through downconversion in rare-earth compounds. *J. Lumin.* **87–89**, 1017–1019 (2000).

2. Kroupa, D. M. et al. Quantum-cutting ytterbium-doped $\text{CsPb}(\text{Cl}_{1-x}\text{Br}_x)_3$ perovskite thin films with photoluminescence quantum yields over 190%. *ACS Energy Lett.* **3**, 2390–2395 (2018).

9:45am **EM1+AP+CPs+MS+PS+QS+SM+TF-TuM-8 Molecular Beam Epitaxy Growth of $\text{InAs}_{1-x}\text{Bi}_x$ on GaSb for Topological Insulating States**, *Merve Baksı, James Rushing, Xikae Xie, Avery Hanna, Larry Qui, Ekow Williams, Paul J. Simmonds*, Tufts University

Incorporation of bismuth (Bi) into III-V semiconductors has attracted significant interest not only for its ability to extend infrared optoelectronic applications across a wide spectral range but also for its potential to induce topologically protected surface states, which could form the foundation for certain quantum computing technologies [1].

Motivated by the small inverted band gap that can be induced in InAs/GaSb quantum wells (QWs) [2], we propose engineering the band structure and inducing the edge states through Bi incorporation into InAs layers. This enhancement is expected to improve robustness against thermal fluctuations, making the material viable for room temperature applications as opposed to the topological HgTe/CdTe QW system with a temperature dependent band gap [3].

Theoretical studies predict that $\text{InAs}_{1-x}\text{Bi}_x$ quantum wells exhibit a topological insulating state when the Bi composition reaches $x \approx 0.15$, with an estimated inverted gap of approximately 30 meV [1]. Given these predictions, InAsBi emerges as a promising candidate for realizing two-dimensional topological insulators (2D TIs). However, achieving such high Bi incorporation remains challenging due to the significant miscibility gap and the limited solubility of Bi in III-V materials [4].

In this work, we investigate the molecular beam epitaxy (MBE) growth of InAsBi on GaSb substrates, focusing on optimizing Bi incorporation and structural quality. By leveraging MBE growth techniques, we aim to systematically control Bi incorporation and assess its impact on electronic and structural properties of InAsBi in reduced dimensions. Our findings will contribute to the advancement of III-V-based topological materials and their potential integration into future quantum devices.

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Plasma Science and Technology

Room 201 ABCD W - Session PS-TuM

Advanced Memory, HARC, and Cryo Etching

Moderators: Jeffrey Shearer, Jeffrey Shearer, TEL Technology Center, America, LLC, Christophe Vallee, UAlbany

8:00am **PS-TuM-1 Optimizing hzfro2 Film Thinning by Plasma Etching for Ferroelectric Memories**, *Vincent Michaud, Christelle Boixaderas, Laurent Grenouillet, Thierry Chevolleau*, CEA-University Grenoble Alps, France

Non-volatile memories are crucial for reducing energy use in modern computing, where most energy is used for data transfer and storage. Ferroelectric Random Access Memories (FeRAMs) retain data without power, lowering energy consumption. The Hafnium-Zirconium Oxide (HZO) has promising ferroelectric properties and its integration is fully CMOS compatible but requires the deposition of a TiN top electrode before the annealing step for crystallization [1] [2]. Recent studies have shown the interest in implementing HZO film thickness lower than 10 nm to reduce voltage operation. However, for such a thickness, the annealing temperature exceeds 400°C which may hamper Back-End Of Line (BEOL) integration [3].

The main goal of this work is to implement HZO thin film with a thickness lower than 10 nm while getting ferroelectric properties for a thermal budget compatible with BEOL integration. The approach is based on the integration of an HZO thin film of 10 nm between the two TiN electrodes (bottom and top) followed by the thermal annealing step to get the ferroelectric properties at a temperature compatible with BEOL scheme. Then, the first step is to etch back the TiN top electrode down to the HZO layer and the second step consists in reducing the thickness of HZO film by plasma etching.

This work involves developing processes to remove the TiN top electrode without damaging the underlying HZO, to optimize plasma etching parameters to thin down the HZO crystallized films below 10nm, while retaining ferroelectric properties. The 10nm thick HZO films deposited by Atomic Layer Deposition (ALD) are partially etched using Inductively Coupled Plasma (ICP) with chlorine-based chemistry. Film thickness is measured by spectroscopic ellipsometry, the surface topography and roughness are analyzed by means of Atomic Force Microscopy (AFM). X-Ray Diffraction (XRD) and X-ray Photoelectron Spectroscopy (XPS) are used to study the impact of plasma etching on the film's structure and surface composition.

The removal of the top electrode is studied using plasma etching or/and wet etching to achieve a selective process to HZO without damaging. The thinning down of the HZO film is based on a parametric study of BCl_3/Cl_2 -based plasma to get an uniform etch process while preserving the crystalline phase with a low surface roughness. Quasi in-situ XPS analysis reveals surface modifications and etching mechanisms, compared to HfO_2 film thinning.

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8:15am **PS-TuM-2 Investigation of Hydrogen and Nitrogen Used as Etching Chemistry or Surface Treatment for Phase-Change Random Access Memory Patterning**, *Benjamin Fontaine*, STMicroelectronics, France; *Christelle Boixaderas, Jérôme Dubois*, CEA/LETI-University Grenoble Alpes, France; *Pascal Gouraud, Arnaud Rival*, STMicroelectronics, France; *Nicolas Posseme*, CEA/LETI-University Grenoble Alpes, France

Phase-change random access memories (PCRAM) have become a crucial technology for data storage, leveraging the resistive tunable properties of a germanium-antimony-tellurium alloy also known as GST. For automotive application requiring high temperature data retention, this material is enriched in germanium resulting in a Ge-rich GST, or Ge-GST [1].

GST is commonly etched using halogen chemistries in inductively coupled plasma reactors [2-3]. The HBr molecule provides a good trade-off in between fast etching and limited film modification. However, it alters the film, leading to the formation of germanium-oxides residues upon air exposure. Recent research focuses on alternative hydrogen and nitrogen-based gases for PCRAM patterning [3].

This study evaluates the effects of H₂ and N₂ plasma etching on Ge-GST films. Both gases have etch rates below 10 nm.min⁻¹ on unpatterned wafers. Atomic force microscopy (AFM) and X-ray Photoelectron Spectroscopy (XPS) reveal that the hydrogen-etched surface is smooth with limited modification, whereas the nitrogen-etched film is rough and significantly altered in stoichiometry. In-situ chemical analysis detected oxygen, fluorine and chlorine contamination after nitrogen etching. To further investigate hydrogen's influence, a specific protocol was developed. Fourier-Transform Infrared Spectroscopy (FTIR) and time-of-flight ion mass spectrometry (ToF-SIMS) indicated substantial hydrogen incorporation in the film post-H₂ etching, likely bounded to germanium and tellurium.

Subsequently, we explored an alternative method to integrate these recent chemistries into Ge-GST patterning. This approach was applied after partial etching of Ge-GST using HBr plasma, as post-etching treatment (PET). The results were consistent with those obtained using the gases as primary etchants. X-ray reflectivity (XRR) measurements showed a negligible GST consumption during both PET. AFM analysis highlighted a smooth surface with hydrogen PET and a rough surface after nitrogen PET. XPS confirmed the preservation of the material stoichiometry plus halogen removal with H₂ PET and the film alteration with N₂ PET.

Finally, hydrogen gas was implemented on memory lines as main etching and PET within the full process including dry stripping, and wet cleaning. Both solutions demonstrated promising results compared to a bromine-based reference process, as evidenced by secondary and transmission electron microscopy (SEM and TEM) coupled with energy dispersive X-ray spectroscopy (EDX).

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8:30am PS-TuM-3 Does the Etching of Exotic Materials or the Implementation of Cryogenic Conditions Call Into Question the Fundamentals of Plasma-Surface Interaction?, *Christophe Cardinaud, Tatiana Mbouja Signe, Felipe Cemin, Hiba Beji*, Nantes Université - CNRS-IMN, France; *Thomas Le Pape*, Nantes Université - CNRS-IMN France; *Aurélien Girard, Cédric Mannequin*, Nantes Université - CNRS-IMN, France

INVITED

Since the early days of plasma etching in the 1970's, mechanisms leading to the etching of a material using a plasma environment have been the subject of numerous studies. The pioneering work of Coburn and Winters set the synergy of interaction between the reactive neutral species and the ion bombardment. In the 1990's numerical expressions of the etch rate versus reactive flux and ion flux were proposed. The most efficient system considers a Langmuir adsorption mechanism for the etchant and an ion flux stimulated desorption mechanism for the etch product. Surprisingly, this model matches experimental data in many situations, where the etching yield is observed to follow a "Langmuir-like" behaviour as function of the ion flux to neutral flux ratio. However, XPS surface analysis, TEM profile and molecular dynamics simulations clearly show that plasma-surface interaction is much more complex. Indeed ion bombardment causes damage to the material; reactive neutrals penetrate the material; so the plasma-surface interaction mechanism is far from being a single-layer process.

In order to better control etched depth, chemical and electrical defects, as well as pattern shape, dimension and surface topography, some etching processes are now moving towards atomic layer etching (ALE) and cryoetching. ALE aims to separate chemical modifications of the surface from the action of ion bombardment, thus breaking with the ion-neutral synergy strategy. Cryoetching aims to increase surface coverage of reactive species at the pattern bottom while promoting passivation mechanisms at the pattern sidewall. Nowadays, it has become a crucial technology in semiconductor manufacturing, as it allows high aspect ratio and selectivity, controllable sidewall profiles and damage in features from the micrometer to the nanometer scale. Recently, it has proved to be of great interest in ALE processes. Behind these technological advances lie several physicochemical mechanisms occurring on the uppermost atomic layers of the cooled surface. Furthermore etching of exotic materials, such as V₂O₃, whose structure may not be as stable as "usual" dielectrics, metal or semiconductors, exhibits strong deviations from the "Langmuir-like" behaviour.

The presentation will briefly review the main findings on the fundamentals of plasma-surface interaction. The evolution of concepts in the case of ALE will also be discussed. Then the physicochemical mechanisms under cryogenic conditions will be presented, with a particular attention to the sample-cooling phase and the effect of residual gases. Finally, the strange case of V₂O₃ will be addressed.

9:00am PS-TuM-5 Charging Dynamics During Pulsed Plasma Etching of High Aspect Ratio Features in Dielectric Materials, *Chenyao Huang¹, Yeon Geun Yook, Yifan Gui*, University of Michigan; *Steven C. Shannon*, North Carolina State University; *Mark J. Kushner*, University of Michigan

During plasma etching of high aspect ratio (HAR) features in dielectric materials (e.g., SiO₂, Si₃N₄, ONO), disparities in the energy and angular distributions (EADs) between positive ions and electrons result in differential charging within the feature. The resulting electric fields within the feature can distort the trajectories of incoming ions resulting in defects and feature distortion. With the aspect ratio of features for memory and logic increasing, there are also increasing concerns of the consequences of charging. A proposed remedy for feature distortion due to charging is the use of pulsed biases. The feature is exposed to different fluxes and EADs of charged species during the on- and off-times during the pulse period. As a result, charging dynamics differ during the on- and off-periods. Net charging likely occurs during the on-portion of the pulsed period when ion energies are high. Discharging likely occurs during the off-portion of the period when ion energies are low, including attracting negatively charged particles into the feature.

In this work we discuss results from a computational investigation of the charging dynamics during pulsed plasma etching of HAR dielectric structures using a 3D voxel-based model, the Monte Carlo Feature Profile Model (MCFPM). MCFPM receives fluxes and EADs of incoming species toward the wafer from a model for plasma properties at the reactor scale, the Hybrid Plasma Equipment Model (HPEM). The MCFPM launches and tracks pseudo-particles representing neutral and charged fluxes towards the surface, and simulates the evolution of the feature and the charging process. The MCFPM includes newly developed algorithms for secondary electron emission processes for electrons and ions. Charging dynamics during pulsed plasma etching of HAR vias will be discussed for fluorocarbon and cryogenic etching of SiO₂ and ONO stacks in multi-frequency capacitively coupled plasmas.

This work was supported by the Department of Energy Office of Fusion Energy Sciences (DE-SC0024545), Samsung Electronics and Lam Research.

9:15am PS-TuM-6 Controlled Cryogenic Silicon Etching Using Pulse-Modulated Platen RF Power, *Zhitian Shi, Nikhil Tiwale, Ming Lu, Chang-Yong Nam*, Brookhaven National Laboratory

Precise control of sidewall profiles and etch anisotropy is critical for high-resolution silicon nanofabrication ¹. In cryogenic reactive ion etching, the interplay of ion flux, radical density, and sheath voltage governs both material removal and sidewall passivation ². Fluorine-based radicals form volatile SiF_x species, while oxygen species stabilize sidewalls via SiO_xF_y passivation layers ³. Platen RF delivery can be pulse-blanked above a threshold, adjusting the duty cycle and influencing etch rate and lateral etching ⁴. Understanding these plasma-surface interactions is essential for transferring nanoscale patterns with high fidelity.

We investigated pulse-blanked RF on cryogenic silicon etching using a 400 nm ZEP hardmask patterned via electron beam lithography (JOEL JBX-6300FS, 500 nm lines, 1 μm pitch, 50% duty cycle). Samples diced from 4-inch wafers were mounted on carrier wafers with Fomblin oil at -100 °C. Etch conditions were constant: chamber pressure 15 mTorr, ICP 2000 W, Platen RF 15 W. Three RF delivery schemes were compared: pulstran 50% duty cycle, sinusoidal-triggered blanking (threshold 4 V, ~70% on-time), and continuous RF (100% on).

Cross-sectional SEM (Figure 1) shows pulsed blanking yields the highest etch rate, with 50% duty cycle increasing ~27% versus unmodulated RF. Sinusoidal-triggered blanking increases ~16%, lower than pulsed, but reduces lateral etching and improves sidewall uniformity. These results indicate that duty-cycle-controlled RF blanking effectively balances etch rate and profile control in cryogenic silicon processing.

Future work will explore the effect of duty cycle, modulating frequency, and phase matching on etch outcomes, and integrate in-situ plasma diagnostics to correlate ion energy distribution with material removal. This approach

provides a pathway toward reproducible, high-fidelity nanoscale pattern transfer, enabling advanced semiconductor, photonic, and quantum device fabrication.

Acknowledgments

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9:30am **PS-TuM-7 Etching Properties of Maskless Oxide/Nitride/Oxide/Nitride (ONON) Stacks with $C_4H_2F_6$ -based Gas**, *Jong Woo Hong*, Nam Il Cho, Geun Young Yeom, Sungkyunkwan University (SKKU), Republic of Korea

The Oxide/Nitride/Oxide/Nitride (ONON; $SiO_x/SiN_x/SiO_x/SiN_x$) stacked structure is commonly used in the 3D vertical architecture of semiconductor memory cells. In the etching of metal contact area of the ONON structure (that is, staircase etching), photoresist (PR) is patterned and repeatedly trimmed over the ONON structure after etching one of the ON layers to form the 3D cells. This layer-by-layer etch process is time-consuming. As a result, a two-step etch method, which involves maskless etching of an ONON stack followed by etching one ON layer at a time with PR trimming, has been adopted using gases such as C_4F_8 or C_4F_6 . However, this two-step approach leads to less ideal etch profiles in the maskless ONON stack in addition to high global warming potentials of C_4F_8 and C_4F_6 gases.

In this study, the etch behavior of maskless ONON stack features using $C_4H_2F_6$ -based gas, which has a lower global warming potential, has been explored and examined its impact on etch characteristics such as etch rate, etch profile, critical dimension (CD) changes, and etch selectivity between SiO_x and SiN_x . The results showed that $C_4H_2F_6$ -based gas achieved the highest etch rates of ONON stack compared to C_4F_6 and C_4F_8 , with an etch selectivity of approximately 1:1 between SiO_x and SiN_x , due to the hydrogen content in the gas. Furthermore, the horizontal CD change was smaller when using $C_4H_2F_6$ compared to C_4F_x -based gases. A thicker carbon-based polymer layer on the sidewall by the etching with $C_4H_2F_6$ also played a crucial role in preserving the top edge shape of the etched maskless ONON stack.

9:45am **PS-TuM-8 Aspect Ratio Resolved Mass Spectrometry for Sticking Probability of Neutral Species in High Aspect Ratio Hole**, *Takumi Kurushima, Takayoshi Tsutsumi, Makoto Sekine, Masaru Hori, Kenji Ishikawa*, Nagoya University, Japan

The demand for microfabrication technology has been increasing as semiconductor devices become three-dimensional (3D) structures. In particular, plasma etching processes for 3D structure are required with high aspect ratio holes and without their shape abnormalities. Simulation-based studies are actively uncovering particle transport and reactions inside the holes, which are difficult to measure them. However, in particle behavior, the sticking probability of radicals on sidewalls is often set based on the number of unpaired electrons, resulting in an approximate value and making it an imprecise parameter. We have developed a novel measurement method to quantitatively determine the sticking probability of neutral species.

The measurements were performed using the appearance mass spectrometry of a quadrupole mass spectrometer (QMS). By varying the aspect ratio of the orifice at the QMS entrance, radicals passed through orifices with different aspect ratios. The fitting results of the experimental and Monte Carlo simulation data lead to a sticking probability. This method is named Aspect Ratio Resolved Mass Spectrometry (ARMS).

From these results, the sticking probabilities of CF , CF_2 , CF_3 , and C_2F_4 were estimated. The ARMS can evaluate the sticking probabilities of neutral species with different mass numbers, contributing to more accurate particle models in simulation-based research.

11:00am **PS-TuM-13 Twisting and Profile Distortion in High-Aspect Ratio Etching Processes**, *Prem Panneerchelvam*, KLA Corporation; *Jin Xie*, KLA Corporation, China; *Chad Huard*, Mark Smith, KLA Corporation
In recent years, the channel hole etching process in 3D NAND manufacturing has experienced significant evolution. Traditionally reliant

on conventional high temperature etching processes with fluorocarbon-based chemistries in pulsed plasmas, the industry has progressively adopted various generations of cryogenic etching to meet the demanding requirements of high aspect ratio structures. The primary challenge in these processes is to preserve the spatial uniformity of the etched profiles despite the extreme aspect ratios involved.

In conventional processes, achieving critical dimension (CD) uniformity was the major focus; however, as the industry transitioned to cryogenic etching, new challenges emerged. Notably, two critical phenomena—twisting and profile distortion—now dominate process variability. Twisting refers to the stochastic deviation of the etching pattern from its intended trajectory, while profile distortion describes the transformation of ideally circular mask openings into non-circular, often triangular, shapes during the etching process.

To elucidate the origins and evolution of these phenomena, we employ ProETCH®, a feature-scale Monte Carlo profile simulator. This tool enables a detailed study of high aspect ratio etch dynamics, offering insights into how stochastic variations lead to twisting and how process conditions contribute to profile distortion. By identifying process windows and parameter adjustments—including changes to plasma properties (IEADs)—this work aims to reduce these effects and improve the overall performance of the etching process.

11:15am **PS-TuM-14 Detection of Etch Products during the SiN_x Etching in a HF Plasma with *In Situ* Mass Spectrometry**, *Xue Wang¹, Md Tanzid Hossain*, Colorado School of Mines; *Prabhat Kumar, Thorsten Lill, Harmeet Singh, Mingmei Wang, Taner Ozel*, Lam Research Corporation; *Sumit Agarwal²*, Colorado School of Mines

We have identified the primary etch products formed during etching of SiN_x with an HF plasma using *in situ* mass spectrometry. As etch products are formed, they can dissociate in the plasma, making direct detection of these species difficult. Due to this complexity, mass spectrometry has been primarily used to detect etch products generated during exposure of the surface to neutrals and ion beams. In other cases, the etch products are detected with a quadrupole mass spectrometer (QMS) downstream of the plasma etcher. While beam studies have provided insight into the basic etch mechanism, the etch products formed in a plasma environment can be different. If the etch products are measured downstream, these species may not represent those that are directly released from the film's surface.

In this work, we identify several etch products during the SiN_x etching in HF plasma using an in-house-built sampling setup for a QMS. As shown in Fig. 1, the QMS housing consists of two stages of differential pumping, and gas phase species were sampled by a skimmer cone positioned ~0.5 cm from surface being etched in the plasma. At an HF pressure of 10 mTorr, this distance is on the order of the mean free path. Additionally, we alternate between self-bias and applied radio-frequency bias at 4 MHz to isolate species that originate directly from the surface. All these designs ensure minimal interaction of etch products with the plasma and other surfaces in the chamber. From the time-resolved mass spectra for SiF_x^+ ($x = 1, 2, 3, 4$) ions during both self-biased and applied-biased etching, we confirmed that SiF_4 is the main etch product. Other fluorosilanes, SiH_xF_{4-x} , were also identified as etch products and the relative ratio of SiF_4 to other fluorosilanes increased with the bias voltage (see Fig. 2). Specifically, we observed that the fraction of SiF_4 increased faster than $SiHF_3$ when a bias was applied, and SiH_2F_2 decreased when switching from the self-bias to the applied-bias condition. This suggests that the reaction pathways change with increasing ion energy during SiN_x etching. Further measurements on N-related etch products indicated that NH_2F is likely an etch product as the QMS signal intensity for NF^+ and NHF^+ increased during etching (see Fig. 3 a, b). Furthermore, the threshold energy for dissociative ionization of NH_2F to NF^+ was ~14.8 eV, which is consistent with the value in the literature (see Fig. 3 c). We confirmed that NF_3 was not a major etch product since we did not detect NF_2^+ and NF_3^+ ions. Finally, the detection of NH_3 remains challenging, primarily due to interference from H_2O adsorbed on the chamber walls.

11:30am **PS-TuM-15 Ion-Enhanced Synergistic Reactions in Cryogenic Plasma Etching with HF-Contained Gases**, *Shih-Nan Hsiao*, Yusuke Imai, Sekine Makoto, Nagoya University, Japan; *Ryutaro Suda, Yuki Iijima, Yoshihide Kihara*, Tokyo Electron Miyagi Limited, Japan; *Masaru Hori*, Nagoya University, Japan

For over half a century, reactive ion etching (RIE) has served as a cornerstone of the semiconductor industry, driving the mass production of

countless wafers daily. Its success lies in the ingenious interplay between reactive chemical gases and energized ions, a synergy that has revolutionized modern fabrication technology. However, as device architectures evolve, conventional RIE struggles to keep pace with the demands of intricate 3D structures featuring tiny dimensions and high aspect ratios. This growing complexity undermines the efficiency of its once-reliable synergistic reactions, resulting in a marked decline in throughput and posing significant challenges to modern fabrication processes. Recently, cryogenic plasma etching containing hydrogen fluoride (HF)-contained species has been reported to address these issues, due to its unique synergistic reactions between ion, surface physisorption species, and material surface [1-3]. To understand the role of ions in synergistic reactions in cryogenic HF plasma, the dependences of bias voltage on etching characteristics and surface structure of the SiO₂ with HF-contained plasmas were investigated. The feeding gas, including CF₄/H₂ and HF, was introduced through a showerhead distributor in the top electrode. *In situ* monitoring techniques, including spectroscopic ellipsometry and attenuated total reflectance Fourier transformation infrared spectroscopy (ATR-FTIR), were used to analyze the surface structure and etching characteristics. The substrate temperature (T_s) was controlled from 20 to -60 °C by circulating a coolant through the bottom electrode. As detailed in the supplemental document, the co-adsorption of HF and H₂O on a cooled substrate introduces a wet-like HF etching mechanism for SiO₂, characterized by an almost zero activation barrier when utilizing pure HF plasma. Interestingly, the etch rate demonstrates an exponential dependence on peak-to-peak bias voltage, deviating sharply from the typical linear relationship observed in conventional chemical-ion sputtering. In the CF₄/H₂ system, fluorocarbon deposition imposes a significantly higher energy threshold for etching. However, a similar trend between etch rate and bias voltage emerges within the high bias voltage regime, further highlighting the complex dynamics of this process.

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11:45am PS-TuM-16 Mechanistic Insights Into Cryogenic Plasma Etching of SiO₂: Temperature, Power and Surface Reaction Dynamics, Yeon Geun Yoak, University of Michigan; *Hyunjae Lee, Sang Ki Nam*, Mechatronics Research, Samsung Electronics Co, Republic of Korea; *Mark J. Kushner*, University of Michigan

In semiconductor manufacturing, overcoming the limitations of high aspect ratio (HAR) plasma etching is critical for continuous scaling of 3D devices. Aspect ratio dependent etching (ARDE) is the slowing of etch rate in as the process proceeds and aspect ratio (AR) increases. Cryogenic etching (CE), cooling the substrate to temperatures as low as -100 C, is emerging as a promising approach for achieving high etch rates and vertical profiles which are less sensitive to ARDE. CE etching of SiO₂ is typically performed in capacitively coupled plasmas (CCPs) whose gas mixtures contain or produce HF. The formation of H₂O through the reaction between HF and SiO₂, and its subsequent condensation, is thought to act as a catalyst which enhances the etch rate. In conventional dielectric etching at ambient temperatures in CCPs, carbon-fluorine reactions dominate the etch process through formation of polymer passivation. The fundamental reaction mechanism responsible for the improved performance of CE has not been clearly defined.

This presentation discusses results from a computational investigation of surface reaction dynamics during the cryogenic plasma etching of SiO₂, employing the Hybrid Plasma Equipment Model (HPEM) and the Monte Carlo Feature Profile Model (MCFPM). Simulations were conducted for dual-frequency CCP reactors using CF₄/H₂/Ar gas mixtures. The temperature-dependent mechanistic differences between cryogenic and room temperature etching were analyzed through parametric analysis of adsorption probability, etch yield, specular reflection of scattering of high energy particles from surfaces, surface diffusion, redeposition, implantation, and condensation. Process parameters including source power (plasma and precursor density) and bias power (ion energy) were also investigated. When compared to conventional dielectric plasma etching, CE increases etch rates by factors of 2-3 with a minimum of bowing or tapering. We found that within estimated ranges of uncertainty, when varying these parameters no single process is uniquely responsible for the improved performance of CE. The improved performance requires synergistic improvements in several key parameters.

This work was supported by Samsung Electronics.

12:00pm PS-TuM-17 Ammonium Fluorosilicate Salt Layer Dynamics during Etching of SiN_x in a HF Plasma and Strategies for Controlling SiN_x to SiO₂ Etch Selectivity, Md Tanzid Hossain, Xue Wang, Colorado School of Mines; *Prabhat Kumar, Thorsten Lill, Harmeet Singh*, LAM Research; *Mingmei Wang*, Lam Research; *Taner Ozel*, LAM Research; *Sumit Agarwal*, Colorado School of Mines

During SiN_x etching with HF plasma, ammonium fluorosilicate (AFS) forms as a transient byproduct, significantly influencing the etch behavior. While AFS formation during SiN_x etching has been known for several decades, the underlying mechanism for its formation and removal remains unclear. In this study, we have used *in situ* attenuated total reflection Fourier-transform infrared (ATR-FTIR) spectroscopy to study the changes in the chemical bonding on the SiN_x surface during reactive ion etching, along with the AFS formation and removal dynamics (Fig. 1). The steady state accumulation of AFS on the SiN_x surface is determined by AFS formation from the etch products and AFS decomposition due to ion bombardment. Prior to etching, the plasma-deposited SiN_x film's surface is terminated with Si-NH_x (x = 1, 2) bonds, and the neutrals in the HF plasma readily react with these species to form AFS, which is apparent from the increase in absorbance for the NH₄⁺ bending and stretching modes at ~1430 cm⁻¹ and ~3000–3300 cm⁻¹, respectively, in Fig. 1. The etch rate of SiN_x is high during the onset of etching due to the abundance of accessible reaction sites. As etching continues, an AFS layer forms, and the etch process likely becomes diffusion-limited as HF, H, and F neutrals have to diffuse through AFS to react with the underlying SiN_x film. Therefore, after the initial burst of AFS formation on the H-terminated SiN_x surface, the process becomes a dynamic competition between ongoing AFS synthesis and removal. On the other hand, on the SiO₂ surface, as expected, there is no AFS residue observed on the surface due to the absence of N in the plasma or in the film. However, the etch rate for SiO₂ is approximately ~3–4 times lower than that for SiN_x under nominally similar conditions. For applications such as etching of alternating stacks of SiO₂ and SiN_x for the fabrication of 3-D NAND memory devices, it is desirable to etch SiN_x and SiO₂ at approximately the same rate. Our initial hypothesis is that AFS formation accelerates the etching of SiN_x compared to SiO₂. Therefore, to promote the etching of SiO₂ we nitrated the surface with an NH₃ plasma to create surface Si-NH_x (x = 1, 2) species (see Fig. 2a). Subsequent HF plasma exposure revealed AFS formation on the SiO₂ surface (Fig. 2b), but it lowered the SiO₂ etch rate compared to the untreated surface. Therefore, to accelerate the etch rate of SiO₂ in a HF plasma, we will explore other process parameters including the average ion energy during etching and surface nitridation, effect of diluents in the gas phase, and the substrate temperature.

Electronic Materials and Photonics

Room 207 A W - Session EM1+CPS+MS+PS+SM+TF-TuA

Advances in Materials and Processes for Devices and Interconnects (FEOL and BEOL)

Moderators: Moon Kim, University of Texas at Dallas, Philip Lee, University of Kentucky

2:15pm EM1+CPS+MS+PS+SM+TF-TuA-1 Carborane-Based Blocking Layers and Plasma Removal Strategies for Area Selective Deposition in Semiconductor Patterning, Raja Sekhar Bale, University of Missouri-Kansas City; *Rupak Thapa*, University of Missouri-Kansas City; *Vamseedhara Vemuri*, Nicholas Strandwitz, Lehigh University; *Anthony Caruso*, *Michelle Paquette*, University of Missouri-Kansas City

Advancing semiconductor scaling to the 2 nm and angstrom-level nodes requires patterning methods that move beyond the resolution, alignment, and cost limits of conventional top-down approaches. Area-selective deposition (ASD) provides a complementary bottom-up strategy, enabling growth only where desired to reduce process steps, minimize defects, and/or improve integration in back-end-of-line (BEOL) fabrication. This work investigates the use of carborane self-assembled monolayers (SAMs) as blocking layers for ASD. Carborane SAMs are thermally stable, mechanically robust, and chemically tunable, making them strong candidates for selective surface modification in semiconductor patterning. Their role is to define surfaces where deposition should be inhibited, while remaining compatible with BEOL conditions. To explore their processing compatibility, the blocking ability of carborane SAMs toward ALD dielectric oxides was studied alongside plasma-based removal. Using CF_4/O_2 and $\text{C}_4\text{F}_8/\text{O}_2/\text{Ar}$ chemistries, we carried out blanket plasma etching of carborane SAMs. This presentation will highlight results on carborane SAM formation, blocking behavior, and plasma response, demonstrating their potential as scalable materials for atomic-scale patterning in next-generation semiconductor manufacturing and their compatibility with advanced BEOL integration.

2:30pm EM1+CPS+MS+PS+SM+TF-TuA-2 Ferroelectricity in Atomic Layer Deposited Wurtzite Zinc Magnesium Oxide $\text{Zn}_{1-x}\text{Mg}_x\text{O}$, Benjamin Aronson, University of Virginia; *Kyle Kelley*, Oak Ridge National Laboratory; *Ece Gunay*, Carnegie Mellon University; *Ian Mercer*, Penn State University; *Bogdan Dryzhakov*, Oak Ridge National Laboratory; *Susan Trolrier-McKinstry*, *Jon-Paul Maria*, Penn State University; *Elizabeth Dickey*, Carnegie Mellon University; *Jon Ihlefeld*, University of Virginia

Ferroelectric wurtzites have garnered interest in the scientific community since first reported in 2019. $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ has shown promise due its low coercive field (2–3 MV/cm) relative to other wurtzites, integrability on flexible polymer substrates, and complementary metal–oxide–semiconductor (CMOS) and back–end–of–line (BEOL) compatible deposition temperatures as low as room temperature. However, the majority of ferroelectric wurtzite thin films – including $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ – have been fabricated using physical vapor deposition (PVD) techniques, which features largely directional growth. Due to the use of high aspect ratio structures in non-volatile memory devices, the ability to conformally deposit ferroelectric wurtzites will contribute to BEOL integration. Atomic layer deposition (ALD) presents an opportunity to overcome this outstanding challenge due to its sequential, self-limiting growth. In this work, $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ thin films with compositions between $x = 0$ and $x = 0.58$ were grown on platinized silicon substrates using plasma-enhanced atomic layer deposition. Films were characterized using X-ray diffraction (XRD), transmission electron microscopy (TEM), and piezoresponse force microscopy (PFM). All films deposited featured a singular out-of-plane c -axis textured wurtzite structure. The c/a ratio decrease with increasing Mg content indicates the increasing structural distortion. Film structure and structural distortions were further reinforced and visualized via TEM. PFM amplitude and phase hysteresis loops demonstrated polarization reversal in the $x = 0.46$ and $x = 0.58$ films. Ultimately, this finding presents opportunities to further mature the $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ processing space in which ferroelectric switching is possible, as well as explore ALD of other ferroelectric wurtzites.

2:45pm EM1+CPS+MS+PS+SM+TF-TuA-3 Harnessing Nitrogen-Rich Interfaces in AlN Ferroelectrics, Ian Mercer¹, Erdem Ozdemir, Chloe Skidmore, Benjamin Debastiani, Kazuki Okamoto, Penn State University; *Sebastian Calderon*, *Elizabeth Dickey*, Carnegie Mellon University; *Susan Trolrier-McKinstry*, *Jon-Paul Maria*, Penn State University

The importance of interface preparation in the nitride semiconductor and thin film community has long been recognized as critical in controlling nucleation and properties. These AlN ferroelectrics are an enticing pathway toward integrated energy-efficient robust non-volatile memory, displaying CMOS chemical compatibility, large polarizations, and BEOL processing. Although this has not been fully realized in the relatively recent nitride wurtzite ferroelectric community, current convention stems from strictly polar systems like GaN and AlN. However, there is a clear opportunity in engineering electrode interfaces in these systems to aid in film nucleation, reduced leakage, and extended fatigue lifetimes. In this work, we discuss the influence of surface nitriding on a variety of relevant substrates prior to film deposition to enhance film texture and electrical properties. Adding the surface nitriding leads to a discussion on whether nitrogen-rich interfaces can compensate for nitrogen vacancies that migrate to electrode interfaces during cycling. By depositing top and bottom metal nitride electrodes, we investigate the benefits in the electrical properties versus metallic electrodes. Reactive RF magnetron sputtering is employed to co-sputter AlN ferroelectrics. X-ray diffraction (XRD) is used to display c -axis texture, while hysteresis (PE), leakage (PUND), and fatigue measurements are used to characterize the electrical properties. Etching/SEM is also used to display partial switching, exploiting the n -polar fast etch in KOH solutions, which helps visualize the effects of nitrogen-rich interfaces. Furthermore, this study reinforces the functionality of interface engineering in AlN ferroelectrics at both the top and bottom electrode interfaces. The importance of this work is that all films in this class may benefit from nitrogen-rich interfaces.

3:00pm EM1+CPS+MS+PS+SM+TF-TuA-4 Selective Etching of GaN Over AlGaIn and Monitoring via Optical Emission Spectroscopy, Michael Thomas, Patrick Wellenius, Spyridon Pavlidis, North Carolina State University

Achieving etch selectivity between GaN and AlGaIn is critical for the repeatable fabrication of enhancement-mode AlGaIn/GaN High Electron Mobility Transistors (HEMTs). The selectivity can be tuned by varying the O_2 content in a Cl_2 -based etch. In this work, we explore the etch process parameter space that affects selectivity and explore how *in-situ* optical emission spectroscopy (OES) can be used as an indicator of chamber and plasma conditions over time.

Two epitaxial structures on sapphire were used. The first is a thin film of GaN (control). The second is a device-relevant AlGaIn/GaN heterojunction with a GaN cap layer. Following photolithography, samples of each type were etched simultaneously in an Oxford Instruments Plasmapro 100 Cobra inductively coupled plasma (ICP) to eliminate run-to-run variation from the selectivity determination. The total etch time was varied by gas composition to keep the HEMT sample etch depth within the AlGaIn front barrier. Etch step heights were measured via atomic force microscopy (AFM) in an Oxford Instruments Asylum Research MFP-3D Origin AFM. Using an OceanOptics USB4000 Spectrometer, OES signals were collected with 1 s integration every 60 s during chamber cleaning and conditioning, and every 30 s during the final etches for each composition.

During initial experiments, the chamber pressure, ICP power, and table RF power were all kept constant at 15 mTorr, 500 W, and 25 W, respectively. The total gas flow was kept constant at 50 sccm, and Cl_2 was further kept constant at 35 sccm. The remaining 15 sccm were split between O_2 and Ar, with three tests being done at 0/15, 2/13, and 4/11 sccm of O_2/Ar respectively. An initial peak selectivity of 3.45:1 was measured with 2 sccm O_2 . The OES signal confirms O_2 emission brightness changes as expected with flow rate. To further improve the selectivity, we will report on the etch characteristics across a wider parameter space, including varying the Cl_2 content of the plasma, the total gas flow rate, the chamber pressure, ICP power and substrate size. Moreover, we explore how the OES's utility can be leveraged to assess the effectiveness of pre-etch chamber conditioning to improve both selectivity and repeatability. The results of this study are expected to boost the yield and performance of AlGaIn/GaN HEMTs.

This technology was primarily supported by the Microelectronics Commons Program, a DoD initiative, under award number N00164-23-9-G059.

Tuesday Afternoon, September 23, 2025

Plasma Science and Technology

Room 201 ABCD W - Session PS1-TuA

Plasmas in Advanced Packaging

Moderators: Catherine Labelle, Intel Corporation, Eric Miller, IBM

2:15pm **PS1-TuA-1 Critical Plasma Processing Steps for Fusion and Hybrid Bonding Applications**, *James Papanu*, Tokyo Electron Corporate Innovation Division / Tokyo Electron Kyushu, Ltd., Japan; *Scott Lefevre, Jeffrey Shearer*, TEL Technology Center America; *Michiko Nakaya*, Tokyo Electron Corporate Innovation Division, Japan; *Yousuke Mine, Yutaka Yamasaki*, Tokyo Electron Kyushu, Ltd., Japan; *Takayuki Ishii*, Tokyo Electron Kyushu, Ltd, Japan; *Christopher Netzbald*, TEL Technology Center America; *Yuji Mimura*, Tokyo Electron Kyushu, Ltd., Japan; *Chikashi Aoyagi*, Tokyo Electron Ltd., Japan; *Ilseok Son, Angelique Raley, Sitaram Aralgud*, TEL Technology Center America

INVITED

Die-to-wafer (D2W) and wafer-to-wafer (W2W) hybrid and fusion bonding are integral to advanced packaging applications. Prior to bonding, for both D2W and W2W approaches, surface preparation is performed to facilitate the bonding process. Surface preparation consists of plasma activation and wet cleaning and hydration process steps. These steps are critical to obtain good interface quality and in turn high yield bonding that is void-free with high bond strength. Plasma surface activation is typically a relatively short, low power process. Nonetheless, the plasma source hardware and process conditions must be optimized to provide sufficient activation without roughening the dielectric layer (fusion and hybrid bonding) or sputtering and/or heavily oxidizing the bond pad Cu (hybrid bonding).

D2W bonding is required for chiplet heterogeneous integration, and also offers the potential for yield improvement by the use of known good die for high bandwidth memory (HBM) and CMOS image sensor (CIS) applications. For D2W bonding, singulated die are bonded directly onto the target wafers. However, the quality of the die singulation process directly impacts the bonding yield. Defectivity levels for traditional saw dicing are too high for high volume D2W manufacturing. As such, advanced singulation techniques, such as plasma dicing are an essential part of the D2W ecosystem. For plasma dicing, there are two approaches, referred to as dice before grind (DBG) and dice after grind (DAG). For DBG, the etching process is performed before wafer thinning. The etching process trenches or grooves the full thickness wafers, and the dies are then singulated during the backgrind thinning process. For DAG, the etching process directly singulates or dices the thinned wafer, landing on a carrier. Consequently, the DBG and DAG have different process requirements and integration challenges.

In this paper, an overview of the fundamental mechanisms, chamber hardware factors, key process parameters, and process integration considerations for surface activation and plasma dicing steps will be presented. In addition, implementation of surface activation plasma for onto bonding cluster tools will be discussed.

2:45pm **PS1-TuA-3 Plasma processing opportunities in the era of Chiplet and Advanced Packaging for AI application**, *Fee Li Lie, Shravana Kumar Katakam, Yann Mignot, Eric Perfecto*, IBM Research Division, Albany, NY

INVITED

The evolution of artificial intelligence (AI) and machine learning (ML) technologies has exponentially accelerated the computing and memory power needed to train AI systems. This leads to larger and larger System on Chip (SoC) dies, some of which are hitting the lithography reticle limit or experiencing area-driven reduction of die yield. There is an additional need for very high bandwidth between processors and large arrays of memory. One emerging solution is to disaggregate large SoC dies into chiplets and re-connect them using advanced packaging techniques. Interconnection between chiplets can occur directly on the package substrate ("2D"), on an interposer ("2.xD"), through stacking of multiple chiplets ("3D"), or potentially a combination of these different technologies depending on application requirements. Die to die interconnect bandwidth and latency are key and we can broadly categorize these connections as lateral or vertical. Lateral interconnection is usually achieved through dual damascene Cu wiring in hard dielectric or plated-up Cu wiring in organic dielectric. Vertical interconnection is usually achieved by using "through" vias, namely Through Silicon Via (TSV), Through Dielectric Via (TDV), or Through Mold Via (TMV), in conjunction with fine pitch micro bump or Hybrid bonding for die to die joining. In this talk, we will discuss plasma processing opportunities in the era of Chiplet and Advanced Packaging, with emphasis on 3D integration with active Si interposers. We will introduce an overview of plasma processes typically used in 3D integration,

and then review scope of needed improvements for some of the critical processes such as TSV RIE, Si thinning, and TSV reveal. Finally, emerging plasma applications for hybrid bonding such as plasma dicing will be introduced.

Plasma Science and Technology

Room 201 ABCD W - Session PS2-TuA

Sustainability and Plasmas

Moderators: Phillipe Bezaud, IMEC Belgium, Sara Paolillo, IMEC Belgium

4:00pm **PS2-TuA-8 Fluorinated Gases in Plasma Etch: Challenges, Accomplishments, and Opportunities**, *David Speed*, GlobalFoundries

INVITED

Plasma etch and chamber clean processes are a primary source of CO₂-e emissions from semiconductor manufacturing processes. Net zero emissions and F-gas phase-out goals bring challenges that require multifaceted solutions, many of which have been the subject of industry efforts for over 30 years. This presentation surveys the principal challenges, accomplishments, and opportunities for achieving reduced CO₂-e emissions from plasma etch processes. Topics to be addressed will include etch process optimization, alternative low-GWP gases, enhanced abatement processes, gas capture and recovery, design integration strategies, and digital twin approaches.

4:30pm **PS2-TuA-10 Kinetic Study of Microwave-Powered, Atmospheric-Pressure Hydrogen Plasma Reduction of Iron Oxide**, *Daniel Ellis¹, Vivek Pachchigar, Jazline Rebollar*, University of Illinois Urbana-Champaign; *Nabiel Abuyazid*, Lam Research Corporation; *Necip Üner*, Middle East Technical University, Turkey; *Ivan Shchelkanov*, Starfire Industries, LLC; *Brian Jurczyk*, Starfire Industries; *Jessica Krogstad, Mohan Sankaran*, University of Illinois Urbana-Champaign

The reduction of iron ore is a key step in steel production. There has been growing interest in applying plasmas to overcome thermodynamic and kinetic limitations with molecular hydrogen as a feedstock. Microwave excitation is of particular interest because of the potential to energy efficiently generate reactive plasma species. Previous studies have been carried out at low (vacuum) pressure or at high temperatures where the contribution of plasma species to the reduction process were not clear.

Here, we studied an atmospheric-pressure, microwave-powered hydrogen plasma for iron oxide reduction. By using a solid-state amplifier to generate the microwave power and a coaxial geometry to transmit the radiation and excite the gas, a plasma jet free from any surface is produced which can be used to treat a material downstream at low temperatures (<400 °C). Using this setup, we treated thin films of iron oxide (hematite) powder to minimize diffusional resistance. The extent of reduction at various process conditions was evaluated by mass loss measurements and X-ray diffraction. The reduction was correlated with plasma properties by optical emission spectroscopy (OES). In particular, the density of hydrogen radicals in the plasma volume was obtained by actinometry and the transport of hydrogen radicals to the iron oxide surface was estimated by a one-dimensional diffusion-advection-recombination model. The surface temperature of the film was obtained by optical infrared pyrometry. All together, we were able to isolate the role of a plasma-activated species, the hydrogen radical, and demonstrate its capability for low-temperature reduction. In addition, a kinetic analysis was performed to obtain an apparent activation energy of ~50 kJ/mol, compared to purely thermal reduction of 92 kJ/mol.

4:45pm **PS2-TuA-11 Transient Assisted Processing (Tap): A Novel Scalable Plasma Processing Approach for Precision Etching and Sustainability in Semiconductor Manufacturing**, *Atefeh Fathzadeh*, KU Leuven and Imec, Belgium; *Philippe Bezaud*, IMEC, Belgium; *Stefan De Gendt*, KU Leuven and Imec, Belgium

New device architectures and computing paradigms require patterning a wide variety of materials with sub-nanometric precision and pattern fidelity, introducing new challenges. For instance, remaining Ga residues after patterning InGaZnO₄ channels cause higher gate leakage in 2T0C DRAM cells¹ or profile imperfections of NbTiN lead to higher power consumption and variations in critical current in Superconducting Quantum Computing (SQC)². Among existing methods, Atomic Layer Etching (ALE) offers excellent precision; however, its high gas consumption and low throughput limit its practicality to ultra-thin layer (≤10 nm) applications.

¹ JVST Highlighted Talk

Tuesday Afternoon, September 23, 2025

These challenges, coupled with environmental concerns of dry etching, have driven interest in developing more sustainable etching approaches.

Transient-Assisted Processing (TAP) provides a breakthrough solution³. TAP is a cyclic process based on reactant transients caused by interrupted gas injection after a short, sustained flow. The dosage, gas injection, and plasma ignition timings ensure an optimal ion-to-neutral ratio and control species formation (Fig1), enhancing process control, pattern fidelity and preserving surface composition. By exploiting the outgassing phenomenon, TAP significantly reduces gas consumption, including environmentally harmful gases, compared to ALE and Reactive Ion Etching (RIE). TAP has also proven effective in precisely cleaning damage-sensitive materials and enabling in situ hard-mask deposition^{4,5}.

This presentation demonstrates TAP's advantages in versatility, scalability, and precision over RIE and ALE for various materials and applications (Fig 2). TAP's sustainability benefits are demonstrated on CMOS BEOL stacks at 24 nm pitch. Additionally, patterning NbTiN/HfZrO/NbTiN capacitors and NbTiN interconnects for SQC at 28 nm pitch showcases TAP's superior pattern control. TAP also provides highly precise control over the etch rate of compound materials, as illustrated by the patterning of IGZO at a 28 nm pitch for 2T0C-based DRAM. The study is also supported by plasma diagnostics, highlighting the underlying plasma mechanisms.

Compatible with as many materials as conventional etching, TAP not only provides better patterning performance and significantly lower consumption of harmful gases, but also achieves precision close to ALE, at a much higher throughput. This makes TAP a more sustainable and higher-performing solution for both current and future applications.

Plasma Science and Technology

Room 201 ABCD W - Session PS3-TuA

Plasma Science Late Breaking Oral Session

Moderators: **Phillipe Bezar**, IMEC Belgium, **Sara Paolillo**, IMEC Belgium

5:00pm **PS3-TuA-12 Atomic Layer Etching of Silicon in Continuous Flow, Pulsed Bias Power Plasmas**, **Vincent Donnelly**, **Jeremy Mettler**, **Qinzheng Hao**, **Mahmoud A. I. Elgarhy**, Dept. of Chem. and Biomolec. Eng, University of Houston; **Heejung Kim**, Semiconductor R&D Center, Samsung Electronics Co., Ltd., Republic of Korea; **Sang Ki Nam**, Mechatronics Research, Samsung Electronics Co., Ltd., Republic of Korea; **Song-Yung Kang**, Semiconductor R&D Center, Samsung Electronics Co., Ltd., Republic of Korea

Atomic layer etching (ALE) provides superior control of etching rate uniformity, but is unacceptably slow for many applications when compared to conventional etching processes. In plasma-assisted ALE required for anisotropic etching, one cycle starts with exposing the substrate to a reactive gas such as Cl₂ for Si or fluorocarbon species for SiO₂, forming an adsorbed layer. The reactive gas flow is turned off and the reactor is purged of this species, usually with an inert carrier gas such as Ar. A plasma is ignited in the inert gas and a substrate bias results in energetic ion bombardment to remove a product layer. The problem with this approach is that the purge step needs to be long enough to reduce the reactive gas to a low enough layer such that no further chemisorption occurs during the ion bombardment step. Otherwise, the self-limiting nature of ALE is compromised. Even with the highest purge gas flow rates, it is difficult to reduce this purge time to less than one second, due at least in part to uptake and release of reactive gases from the chamber walls. Here we take a different approach to plasma ALE. With a constant inductively-coupled plasma (ICP) power, a continuous flow of reactant gas (Cl₂) was maintained in a continuous high flow of Ar carrier gas. Etching occurred during periodic substrate bias power periods (-75 DC self-bias voltage) of 0.2s to 0.5s. Between non-ALE continuous etching at too high a Cl₂ partial pressure, and a rate approaching physical sputtering at too low a Cl₂ partial pressure, an "ALE window" was found at low Cl₂ partial pressures where the chlorination rate during 1s periods with no bias power formed a SiCl_x layer that was sputtered away during the substrate bias period at a rate that was faster than the rate at which the surface could re-chlorinate. Under these conditions, an etching rate of ~0.2nm/cycle, or about 1 monolayer of Si was achieved. Optical emission spectroscopy and a simple Langmuir-Hinshelwood model were used to monitor SiCl desorption during the ALE cycles and provided further insights into the etching mechanism. This work was supported in part by Samsung Electronics (project code IO240308-09220-01).

5:15pm **PS3-TuA-13 Correlation of Optical Emission Spectroscopy Line Ratios with Deposition Rate and Refractive Index of Silicon Nitride Films in Plasma Enhanced Chemical Vapor Deposition**, **YOUNGJU KO**, **HYEONJIN CHOI**, **JINMYEONG KIM**, Sungkyunkwan University (SKKU), Republic of Korea; **NAMGUN KIM**, Samsung Electronics Co., Republic of Korea; **HEEYEO CHAE**, Sungkyunkwan University (SKKU), Republic of Korea

Optical emission spectroscopy (OES) is common non-invasive method for monitoring plasma in semiconductor manufacturing and analyzes emitted light without disturbing the plasma. Quantitative understanding of plasma states from OES peak intensities is still challenging. In this work, the deposition rate and refractive index of silicon nitride (SiN_x) deposited using trisilylamine (TSA), NH₃ and N₂ gas were predicted using OES analysis in plasma enhanced chemical vapor deposition (PECVD). The four dominant peaks of 337 nm (N₂ second positive system), 391.2 nm (N₂⁺ first negative system), 656 nm (H_α Balmer line), and 486 nm (H_β Balmer line) were selected, and the correlation between the deposition rate and intensity ratios of I_{N2⁺}/I_{N2} and I_{Hα}/I_{Hβ} was investigated. The I_{N2⁺}/I_{N2} was found to be strongly correlated with the deposition rate with coefficient of determination (R²) of 0.85 and mean absolute percentage error (MAPE) of 3.66%. This strong correlation is attributed to the fact that the ratio represents the variation of electron temperature, which increases molecular dissociation and ionization in plasma. However, the refractive index was poorly correlated with the I_{N2⁺}/I_{N2} and I_{Hα}/I_{Hβ} line ratios, and the intensity ratios of I_{NH}/I_{N2} and I_{SiH}/I_{N2} were suggested from 336 nm (NH), 414.2 nm (SiH), and 337 nm (N₂) peaks as indicators representing the relative radical density of NH and SiH radicals. These line ratios were derived because they have similar overlap of excitation cross sections with electron energy distribution function (EEDF) in typical inductively coupled plasmas (ICP). The derived I_{SiH}/I_{NH} ratio showed a strong correlation with the refractive index, as the atomic composition of N and Si in the film is directly influenced by NH and SiH radicals in plasmas. The refractive index with I_{NH}/I_{N2} and I_{SiH}/I_{N2} line ratios showed high accuracy with R² of 0.95 and MAPE of 0.27%. This work demonstrated that the OES intensity ratio proposed as I_{N2⁺}/I_{N2} and I_{SiH}/I_{NH} can effectively predict deposition rate and refractive index in SiN_x PECVD.

5:30pm **PS3-TuA-14 Particle-in-Cell Simulations of Low Pressure Magnetized Plasma**, **TAARESH TANEJA**, Applied Materials Inc.; **SHAHID RAUF**, Applied Materials, Inc.; **PRASHANTH KOTHNUR**, Applied Materials Inc.

Low pressure magnetized plasmas (e.g., magnetrons) are widely used for metal deposition in the semiconductor industry. This study focuses on kinetic phenomena important in these plasma discharges. In particular, the generation and propagation of waves of electron and ion densities are studied. One-dimensional Particle-In-Cell (PIC) simulations with Monte Carlo Collisions (MCC) of argon plasma are used for this investigation. The simulations were conducted across a range of pressures (1–10 mTorr), ion masses (10 amu – 160 amu), and external magnetic field strengths (peak ranging from 200 G to 400 G) to explore the influence of these parameters on the plasma dynamics. The plasma was confined between a cathode and an anode, with a magnetic field applied perpendicular to the simulation axis that exponentially reduces from the cathode to the anode.

Distinct wave-like structures were observed propagating from the anode towards the cathode under various conditions. These waves exhibited clear dependence on the magnetic field strength and ion mass, suggesting a magnetically driven mechanism. Detailed analysis of the wave frequency and phase velocity revealed characteristics consistent with ion cyclotron waves, including their scaling with the ion cyclotron frequency and propagation behavior in magnetized plasmas. However, these ion and electron density waves are not found to be in perfect resonance with the ion cyclotron mode. The frequency was found to also scale inversely with the square root of the ion mass, suggesting ion acoustic wave contribution. Moreover, the wave amplitude was found to rise in the direction of propagation, suggesting a non-linear, oscillation growing mechanism. This change in amplitude was much more pronounced at higher pressures, such as 10 mTorr.

The identification and analysis of these waves provide insight into ion transport and energy redistribution in magnetized low-pressure discharges, which are critical to optimizing sputtering efficiency and uniformity in physical vapor deposition processes. The results demonstrate the capability of PIC-MCC simulations to capture kinetic and magnetic field-induced transport effects that are often very crudely approximated in fluid models. These findings contribute to a deeper understanding of plasma behavior in magnetron systems and may inform the design of more efficient and controlled deposition environments.

Tuesday Afternoon, September 23, 2025

5:45pm **PS3-TuA-15 Effect of Nitrogen Addition on Electron Density and Uniformity Enhancement in Oxygen Plasma, Yeongjae Jeong, Chin-Wook Chung**, Hanyang University, Korea

In oxygen plasmas, the formation of negative ions leads to a reduction in electron density at the discharge center and degrades the overall discharge uniformity. This study investigates the effects of adding small amounts of nitrogen to oxygen discharges, focusing on changes in electron density and spatial uniformity. Experiments were carried out in a 13.56 MHz inductively coupled plasma at 200 mTorr under varying nitrogen admixture ratios. The electron energy probability function (EEPF) was measured radially using an RF-compensated Langmuir probe. The results show that the addition of nitrogen increases the central electron density by up to 42% and improves discharge uniformity. This phenomenon is attributed to vibrational excitation of nitrogen molecules, which acts as a competing energy loss channel for low-energy electrons, thereby suppressing the formation of negative oxygen ions and reducing electron loss. In this study, we define a parameter termed electron retention efficiency to quantify this effect and analyze its correlation with electron density enhancement. The results indicate that higher electron retention efficiency leads to a more significant increase in electron density, demonstrating that minor nitrogen addition can be an effective method to control electron density and uniformity in oxygen plasmas.

Atomic Scale Processing Mini-Symposium

Room 206 A W - Session AP+PS+TF-WeM

Thermal and Plasma-Enhanced Atomic Layer Deposition

Moderator: Austin Minnich, California Institute of Technology

8:00am **AP+PS+TF-WeM-1 High-Temperature Thermal ALD of SiO₂ Using Chlorosilane and Aminosilane Precursors: A Comparative Study**, *Okhyeon Kim, Tanzania Chowdhury, Changgyu Kim, Hye-Lee Kim*, Sejong University, Republic of Korea; *Jae-Seok An, Jung Woo Park*, Hansol Chemical Co., Ltd., Republic of Korea; *Won-Jun Lee*, Sejong University, Republic of Korea

As the number of layers in three-dimensional vertical NAND memory devices continues to increase, the conformal deposition of high-quality SiO₂ films in high-aspect-ratio (HAR) structures at high temperatures becomes increasingly critical. Atomic layer deposition (ALD) is the ideal technology for this application, offering atomic-level thickness control and excellent conformality. However, most existing studies on SiO₂ ALD have focused on low-temperature plasma-enhanced ALD processes, which are inadequate for producing high-quality films in HAR structures. In contrast, high-temperature thermal ALD of SiO₂ films remains underexplored. In this work, we investigated high-temperature (>600 °C) thermal ALD of SiO₂ using chlorosilane and aminosilane as Si precursors and compared the performance of silicon precursors. Density functional theory (DFT) calculations were first performed to evaluate the precursors based on their thermal stability. Next, the maximum ALD temperature was determined experimentally based on self-limiting behavior and confirmed by step coverage analysis in HAR patterns. Film composition and impurity levels were analyzed by X-ray photoelectron spectroscopy and dynamic secondary ion mass spectroscopy. Stoichiometric SiO₂ films were deposited using both chlorosilane and aminosilane precursors, but pure ALD processes were possible at higher temperatures with chlorosilane precursors due to their better thermal stability. Chlorosilane precursors also resulted in lower impurity levels in the film due to their simpler molecular structures, which is consistent with the better electrical properties and wet etch resistance observed. This study combines theoretical and experimental results to provide a basis for advancing high-temperature thermal ALD processes of SiO₂ and related materials.

8:15am **AP+PS+TF-WeM-2 Catalyzed Molecular Layer Deposition of Methylene-Bridged Silicon Oxycarbide and the Effect of Annealing on Molecular Structure and Electrical Properties**, *Man Hou Vong, Seoyeon Kim, Michael Dickey, Gregory Parsons*, North Carolina State University

Silicon oxycarbide (SiOC-H) is a low-k dielectric material capable of minimizing parasitic capacitance between interconnects, thereby lowering the signal delay. As feature nodes in integrated circuits continue to shrink, deposition processes that offer precise control over film thickness and conformity are increasingly critical. Molecular layer deposition (MLD), a vapor deposition technique that deposits molecular layers via self-limiting surface reactions driven by sequential reactant exposure, offers a promising route to meet these demands. Previous studies have demonstrated the feasibility of MLD for methylene-bridged (Si-CH₂-Si) SiOC-H using bis(trichlorosilyl)methane (BTCSM) as the precursor and water as the oxidant at moderate temperatures (< 100°C). However, the reported growth rate was limited despite the high reactant exposure. We hypothesize that the limited growth arises from the inefficient direct reaction between the Si-Cl on BTCSM and the Si-OH on the substrate surface. Herein, we introduce a catalyst to overcome the growth limitation in SiOC-H MLD using BTCSM and water. The results show that incorporating catalyst in MLD of SiOC-H at 50°C increases the growth rate by more than ten times under identical reactant exposure. Furthermore, upon annealing at temperatures from 250°C to 550°C, the Si-CH₂-Si bridges undergo a transformation into terminal methyl groups (Si-CH₃) via reaction with adjacent Si-OH groups. This transformation increases steric hinderance within the film compared to methylene bridges, reducing the film density and ultimately lowering the permittivity of the films. Overall, the findings in this work provide insights into the role of the catalyst in SiOC-H MLD and highlight its potential for enhancing deposition efficiency for scalable manufacturing in advanced microelectronics fabrication.

8:30am **AP+PS+TF-WeM-3 The Effect of Precursor Choice and Process Temperature on the Properties of ALD Films**, *Theodosia Gougousi, Nimarta Chowdhary*, UMBC

Precursor choice and process temperature play a critical role in determining the properties of thin films deposited by Atomic Layer Deposition (ALD). In this study, we examine the impact of deposition temperature on the properties of ALD metal oxide films grown using amide-based precursors:

tetrakis dimethyl amino titanium (TDMAT) and tetrakis dimethyl amino hafnium (TDMAHF) with water as the oxidizer.

We observe distinct differences between the two precursors. For the Ti process, we find a significant influence of temperature on phase formation and nitrogen incorporation into the films. Films deposited at 100°C crystallize in the anatase phase after inert annealing, while those deposited between 150–300°C transition to the rutile phase. At 350°C, films exhibit mixed phases that vary with thickness. Additionally, films deposited at temperatures above 200°C incorporate oxynitride bonding, significantly affecting both their linear and nonlinear optical properties and electrical conductivity. These variations are most pronounced between 200 and 275°C, a temperature range commonly considered within the "ALD window" for this process.

In contrast, for the Hf process, we do not observe any nitrogen incorporation in the films even at 400°C and the optical and electrical properties of the films are consistent across deposition temperatures. Our findings reveal previously unreported reaction pathways that significantly influence the optical and insulating properties of TiO₂ ALD films. Furthermore, we highlight significant differences in the behavior of precursors from the same family emphasizing that extrapolating properties from one materials system to another can be misleading.

This study provides significant insights into the temperature-dependent behavior of ALD-grown TiO₂ and HfO₂ films, highlighting previously unreported reaction pathways. These findings offer valuable guidance for optimizing film properties in optoelectronic applications and underscore the importance of precise precursor selection in ALD processes.

8:45am **AP+PS+TF-WeM-4 Microwave Enhanced Atomic Layer Deposition (MW-ALD) of HfO₂**, *Jessica Haglund, John Conley Jr.*, Oregon State University

Though beneficial for many applications, the low temperatures typical of ALD can result in residual impurities from unreacted precursors. This can lead to degraded electrical, physical, and optical properties. To improve film quality, post deposition annealing (PDA) can be used. However, the high temperatures necessary for PDAs can exceed thermal budgets, especially in back end of line processing. It has been demonstrated that *post-deposition* microwave annealing can improve film quality and result in lower process temperature.¹ An alternate way to improve film quality is energy enhanced ALD (EE-ALD), in which energy is added during the ALD cycles. Previously, in-situ rapid thermal anneal, plasma, and UV treatments have been added to ALD cycles to drive impurities from films during deposition.²⁻⁶ We have recently introduced in-situ microwave enhanced ALD (MW-ALD) using Al₂O₃.⁸ Here we discuss low temperature MW-ALD of HfO₂.

A custom MKS microwave generator and helical antenna were integrated into a Picosun R200. HfO₂ was deposited at 150 °C using 100 TEMA-Hf/N₂/H₂O/N₂ ALD cycles of 1/120/0.2/120 sec. A 30 s 400 W microwave (MW) pulse (without plasma generation) was used during either the TEMA-Hf or the H₂O purge. Film thickness and refractive index were analyzed using a mapping Film Sense FS-1 ellipsometer. MW pulses during the H₂O purge had minimal impact on film thickness and refractive index. However, the same MW pulse during the TEMA-Hf purge resulted in a ~50% increase in thickness and an increase in refractive index. This is consistent with our work on MW-ALD of Al₂O₃ which found an increase in film quality when the pulse was applied during the TMA pulse as compared to the water pulse.⁸ Additional electrical data will be presented as well as results for depositions at 250 °C.

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9:00am **AP+PS+TF-WeM-5 In Situ Studies of Ald Hf_{0.5}Zr_{0.5}O₂ by Spectroscopic Ellipsometry and Reflection Absorption Infrared Spectroscopy**, *Stijn van der Heijden, Alex Neefs, Erwin Kessels, Bart Macco*, Eindhoven University of Technology, Netherlands

Ferroelectric Hf_{0.5}Zr_{0.5}O₂ (HZO) is widely recognized as a leading material for next-generation non-volatile memory technologies, offering excellent scalability and seamless integration with CMOS processing. We have developed an atomic layer deposition (ALD) process for HZO using metalorganic precursors—HfCp(NMe₂)₃ and ZrCp(NMe₂)₃—in combination with ozone as the oxygen source. This process enables controlled deposition with precise Hf:Zr stoichiometry and uniform film growth under optimized conditions.

In situ spectroscopic ellipsometry (SE) on an Oxford Instruments FlexAL was used extensively during process development to monitor film growth in real time and to extract growth-per-cycle data. Additionally, full TiN/HZO/TiN capacitor stacks were fabricated in a single ALD sequence within the same reactor, allowing us to track film evolution throughout the stack formation. This provided detailed insight into the nucleation behavior and the formation of interfacial layers.

To gain a deeper understanding of the surface chemistry, we employed *in situ* reflection absorption infrared spectroscopy (RAIRS) using a home-built reactor. The RAIRS analysis revealed that formate groups, generated during the ozone pulse, act as active surface sites for precursor adsorption in both the HfCp(NMe₂)₃ and ZrCp(NMe₂)₃ processes.

Finally, we correlate the findings from SE and RAIRS with the electrical performance of the TiN/HZO/TiN capacitors, offering an integrated view of how surface chemistry and film nucleation influence ferroelectric behavior.

9:15am **AP+PS+TF-WeM-6 Thermal Stability of HfO₂ by Incorporating Al₂O₃ in a MIM Capacitor by 200 nm Batch-ALD**, *Partha Mukhopadhyay*, Tokyo Electron America; *Ivan Fletcher, Zurriel Caribe, Anton deVilliers, Jim Fulford*, Tokyo Electron America, USA

This work investigates the thermal stability of HfO₂-Al₂O₃ laminated high-k dielectrics deposited by the high-volume batch atomic layer deposition (ALD) method. At higher crystallization temperatures HfO₂ converts from amorphous to polycrystalline and induces nonuniformity in film thickness. The incorporation of Al₂O₃ into the HfO₂ film forms an HfAlO alloy which presents excellent thermal stability compared to pure HfO₂ when annealed at 650°C. Cross-sectional TEM, SIMS and XPS profiles demonstrate the interfacial reaction of these ultra-thin layers where the core-level energy states, Hf4f and Al2p peaks showed a shift to higher binding energy from those of pure HfO₂ upon Al₂O₃ incorporation (Fig. S2). It is mainly because the Al covalence changes the bonding characteristics and HfO₂ becomes more ionic, therefore, the dissociation of the alloyed film is effectively suppressed compared to a pure HfO₂ film, indicating an enhanced thermal stability of HfAlO. The fabricated MIM capacitor of low Al-content Hf_{0.69}Al_{0.31}O alloy exhibits a higher capacitance density (C_pD) of 12.46 fF/μm², ~29% better than HfO₂ and dielectric constant of κ>22 than HfO₂. The present research indicates a small amount of Al (0.31) incorporation in HfO₂ extends its quantization temperature due to stabilizing its crystal phase by reducing oxygen vacancies and traps. It remarkably improved electrical characteristics under thermal stress compared to broken-down HfO₂ capacitors under annealing (Fig. S3). While a higher Al content Hf_{0.44}Al_{0.56}O alloy shows excellent thermal stability while possessing 68% higher κ than an Al₂O₃ capacitor. It also demonstrated the highest breakdown voltage (E_{BD}) of 8 MV/cm and low leakage among the samples. After annealing the degradation of E_{BD} of the HfO₂ capacitor is nearly 94% while the HfAl_{0.31}O capacitor faces only 19% (Fig S4). These thin multilayer alloys show excellent relative capacitance variation over the voltage with high C_pD, κ-value, low leakage of 10 nA/cm²@3MV/cm, suitable for higher thermal budget BEOL, and interposer process integration for various high bandwidth RF and low-cost memory applications with smaller chip area.

9:30am **AP+PS+TF-WeM-7 Highly Crystalline ZrO₂ Films under 2 nm by Atomic Layer Modulation**, *Wonjoong Kim*, Incheon National University, Republic of Korea; *Ngoc Le Trinh*, Incheon National University, Viet Nam; *Bonwook Gu*, Incheon National University, Republic of Korea; *Byungha Kwak*, Ajou University, Republic of Korea; *Hyunmi Kim, Hyeongkeun Kim*, Korea Electronics Technology Institute, Republic of Korea; *Youngho Kang*, Incheon National University, Republic of Korea; *Il kwon Oh*, Ajou University, Republic of Korea; *Han-Bo-Ram Lee*, Incheon National University, Republic of Korea

As the dimensions of silicon-based devices continue to shrink, achieving both high capacitance and low leakage current becomes increasingly challenging. In particular, the corresponding reduction in thin film thickness

makes it difficult to preserve critical physical properties, including crystallinity, thermal stability, and electrical performance. In this work, we investigated yttrium-doped zirconium oxide (YZO) thin films fabricated using atomic layer modulation (ALM), a technique based on atomic layer deposition (ALD). In the ALM process, the surface is sequentially exposed to two precursors with an intervening purging step between each exposure, followed by a reaction with a counter-reactant, resulting in the growth of the YZO film within a single atomic layer. The ratio of Y to Zr in the ALM film is determined by the steric hindrance and chemical reactivity of the precursors with the surface. To design and interpret the experimental process, two theoretical approaches—density functional theory (DFT) and Monte Carlo (MC) simulations—were employed to examine the precursor interactions and their impact on film composition. In ALM films, Y atoms are located closer to Zr atoms, leading to the formation of Y–O–Zr bonds in both the lateral and vertical directions within several atomic layers. Consequently, the ALM film requires a lower energy barrier for diffusion to form the YZO crystalline phase, which enhances film density and improves crystallinity. As a result, YZO films deposited via the ALM process exhibit approximately 250 times lower leakage current density compared to the conventional YZO films fabricated using the ALD under a thickness of 2 nm. This key finding highlights that YZO films prepared by ALM achieve both an increased dielectric constant and reduced leakage current density at low thicknesses, demonstrating their potential as promising materials for future silicon device applications.

9:45am **AP+PS+TF-WeM-8 Influence of Molecular Structure on Ruthenium Deposition: An *in Situ* Study Using Simultaneous Spectroscopic Ellipsometry and Quadrupole Mass Spectrometry**, *Terrick McNealy-James*, University of Central Florida; *Xin Kang*, University of Florida, Gainesville; *Luis Tomar*, University of Central Florida; *Johnathon Johnson*, University of Florida, Gainesville; *Novia Berriel, Taylor Currie, Titel Jurca*, University of Central Florida; *Lisa McElwee-White*, University of Florida, Gainesville; *Parag Banerjee*, University of Central Florida

Ruthenium (Ru) with its low bulk resistivity and high work function has emerged as a promising metal for future interconnect technology. Numerous Ru complexes with different ligands have been studied to refine chemical vapor deposition (CVD) and atomic layer deposition (ALD) processes and improve film structure, property and performance. These include molecules such as, bis(cyclopentadienyl)ruthenium [RuCp₂], tris(2,2,6,6-tetramethyl-3,5-heptanedionato)- ruthenium [Ru(thd)₃] and η⁴-2,3-dimethylbutadiene ruthenium tricarbonyl [Ru(DMBD)(CO)₃].¹⁻³

Here we investigate the ALD process characteristics of Ru thin films from (η⁴-diene)Ru(CO)₃ complexes and resulting film properties. Three molecules are chosen i) η⁴-isoprenylruthenium tricarbonyl, ii) (η⁴-1,3-butadiene)ruthenium tricarbonyl and iii) (η⁴-1,3-cyclohexadiene)ruthenium tricarbonyl; with the rationale of studying the effect of changes to the ligand motif on the film growth characteristics and resulting properties. Furthermore, by employing simultaneous *in situ* spectroscopic ellipsometry (SE) and quadrupole mass spectrometry (QMS), we disambiguate the physical growth mechanisms and chemical reactions occurring at the substrate surface. The resulting Ru film properties are analyzed *ex situ* using x-ray photoelectron spectroscopy (XPS), x-ray diffraction (XRD) and four-point probe resistivity measurements.

Our *in situ* SE measurements show that, in all cases, deposition occurs for temperatures ≥ 160 °C. No self-saturation in growth behavior is observed. This aligns with QMS data which suggests that all Ru complexes undergo spontaneous dissociation reaction on the substrate surface. The water half-reaction plays no relevant role in promoting deposition. XPS and XRD analyses reveal that all films consist of a Ru/RuO_x mixture in line with high film resistivity. These results highlight the limited role of ligands in controlling the ALD / CVD film growth characteristics of diene-Ru(CO)₃ complexes with H₂O as a co-reactant.

11:00am **AP+PS+TF-WeM-13 Study on the Thermal Decomposition Behavior of Mo(Co)6 as a Precursor for Mo-ALD**, *Soken Obara, Souga Nagai, Jun Yamaguchi, Noboru Sato, Naoki Tamaoki, Atsushi Tsukune, Yukihiko Shimogaki*, The University of Tokyo, Japan

As miniaturization advances in state-of-the-art semiconductor devices, interconnect resistance becomes increasingly problematic. Atomic layer deposition (ALD) of molybdenum (Mo) is gaining attention as a potential next-generation interconnect technology to replace conventional Cu and W. Although Mo precursors such as MoCl₅ and MoO₂Cl₂ are commonly used, they present significant drawbacks, including the need for high processing temperatures (~600 °C) and the presence of halogens. In this study, we investigated the thermal decomposition and adsorption behavior of

Mo(CO)₆, a halogen-free precursor capable of deposition at lower temperatures, to evaluate its suitability for Mo-ALD.

Using an ALD system equipped with a bubbling delivery mechanism, we deposited Mo films on Si substrates with 100 nm thermal oxide at temperatures ranging from 130 to 175 °C, using Mo(CO)₆ and NH₃ as precursor and reactant, respectively. Figure 1 shows the ALD process sequence and growth-per-cycle (GPC) as a function of temperature, revealing a steep increase in GPC above 150 °C. As shown in Fig. 2, the precursor pulse time dependence at 145 °C deviates from the ideal ALD self-limiting behavior, indicating a CVD-like growth mechanism.

To investigate the thermal decomposition characteristics of Mo(CO)₆, film deposition cycles were performed at 175 °C using only Mo(CO)₆ and purge gas, without NH₃. As shown in Fig. 3, film formation was observed with a 4-second purge, diminished with an 8-second purge, and disappeared completely with a 14-second purge. This suggests that physisorbed species were gradually removed by purge, thereby suppressing film formation. To determine whether chemisorbed species remained on the surface, a subsequent ALD process with NH₃ was performed after an 8-second purge. As shown in Fig. 4, Mo film growth was observed in the downstream region, suggesting that chemisorption persisted even after the longer purge duration.

Step coverage results are shown in Fig. 5. Under ALD conditions at 145 °C and 175 °C with a 4-second purge, step coverage was 100% and 91%, respectively. However, under the 175 °C condition with an 8-second purge, the step coverage exceeded 165%, indicating thicker deposition at the bottom. This result is attributed to residual physisorbed species accumulating at the feature bottom, leading to enhanced local film growth. These findings demonstrate that bottom-up filling can be achieved by tuning the purge time of the precursor.

11:15am AP+PS+TF-WeM-14 Nucleation Enhancement and Growth Modification in Co-ALD via Pd activation, Yubin Deng, The University of Tokyo, Japan, China; *Souga Nagai, Jun Yamaguchi, Yuhei Otaka, Noboru Sato, Naoki Tamaoki, Atsuhiko Tsukune, Yukihiro Shimogaki,* The University of Tokyo, Japan

With the continued downscaling of ULSI technologies to the 3 nm node, Cu interconnects demand increasingly thinner liner/barrier layers that can ensure reliable performance under aggressive miniaturization. Previous studies have demonstrated that 1-nm-thick Co(W) films exhibit excellent Cu diffusion barrier properties [1]. However, the critical challenge remains achieving ultrathin, continuous films with precise thickness control. In this context, ALD is considered the most promising technique, offering conformal and selective growth suitable for high-aspect-ratio structures. Importantly, fabricating thinner films via ALD requires higher nucleation densities, which can be promoted by Pd activation. The catalytic properties of Pd enhance precursor adsorption and subsequent surface reactions, thereby improving nucleation. In this study, we systematically investigated the impact of Pd activation on the nucleation behavior and morphological evolution of ALD-Co films.

All samples were prepared on Si substrates with a 300-nm-thick thermally grown SiO₂ layer and were cleaned using ethanol and APM. Two Pd activation methods were employed. The conventional wet method involved immersion in a colloidal Sn/Pd solution (0.6 mM PdCl₂, 30 mM SnCl₂, 0.35 M HCl) at 40 °C for 5 min (Fig. 1), followed by a 3 min rinse in 1 M HCl to remove residual Pd and byproducts, and subsequent drying. Alternatively, Pd activation was performed using ALD (Fig. 2) at 200 °C for 400 cycles, employing palladium(II) hexafluoroacetylacetonate (Pd(hfac)₂) as the precursor and aqueous formalin (HCHO) as the reducing agent, with N₂ as the carrier and purge gas. Following Pd activation, Co films were deposited via ALD at 150 °C for 500 cycles (Fig. 3), using dicobalt hexacarbonyl tert-butylacetylene (CCTBA) and H₂ as the precursor and reactant, respectively.

In the wet method, Pd loading was controlled by varying solution concentration and activation time. While in Pd-ALD, it was precisely adjusted by tuning the precursor pulse count per cycle (supply time). As shown in Fig. 4(a), the wet method failed to deposit sufficient Pd on thermal SiO₂, even with extended activation (50 min) and highly concentrated solutions (20×). In contrast, Pd-ALD enabled fine control over the Pd amount, as shown in Fig. 4(b). Figure 5 presents the effects of Pd loading on Co nucleation and morphology. Increased Pd loading resulted in smaller and denser Co nuclei (~9 nm, ~1.1 × 10¹² cm⁻²) and enhanced Co deposition. To achieve uniform 1 nm-thick Co films, further optimization of the Pd-ALD process is necessary to reach the target nucleation density (~10¹⁴ cm⁻²).

References

Wednesday Morning, September 24, 2025

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11:30am AP+PS+TF-WeM-15 Process-Structure-Properties of Atomic Layer Deposited Niobium Nitride and Evolution of Strain with Plasma Chemistry, Neeraj Nepal, Joseph Prestigiacomo, Maria G Sales, Peter M Litwin, Vikrant J Gokhale, Virginia D Wheeler, Naval Research Laboratory

Niobium nitride (NbN) has exceptional physical, chemical, and electrical properties that can be utilized in a range of applications such as gate metal, superconducting qubits and detectors (T_c ~9-17 K [1]), RF antennas, resonators, and Cu interconnect diffusion barriers. For all these applications, a low temperature growth process with wafer scale uniformity, conformality, and subatomic thickness control is highly desirable. Atomic layer deposition (ALD) provides a path towards integration of NbN at lower temperatures with control over the desired properties. Most reported thin plasma-enhanced ALD (PEALD) NbN films [2-3] to date are either amorphous or polycrystalline. In this talk, we report on highly oriented single phase, PEALD NbN (111) films and discuss the evolution of strain with plasma chemistry.

ALD NbN films were deposited on resistive Si and c-sapphire in a Veeco Fiji Gen2 ALD reactor using (t-butylimido)tris(diethylamido)niobium(V) (TBTDEN) and N₂/H₂ plasma precursors. Similar to previous reports [2], TBTDEN required a boost to enable growth. Growth windows and film morphological, structural, and electrical properties were optimized for TBTDEN temperature (80-100°C), TBTDEN boost (1-2s), TBTDEN pulse (1.5-2.0s), plasma pulse (20-30s), H₂/N₂ ratio (1.5-12.5), and temperature (150-400 °C). Optimum growth parameters (TBTDEN = 100°C, TBTDEN boost = 1.5s, TBTDEN pulse = 2s, and H₂/N₂ = 60/20sccm) yielded an ALD window from 250-300°C with a growth rate (GR) of ~0.5Å/cy. While GR was almost constant for N₂ ≥ 20 sccm, room temperature resistivity (ρ_{RT}) increased linearly with N₂ flow. High-resolution XRD scans show 1st and 2nd order (111) NbN peaks. Lattice constants obtained from XRD show that strain changes from compressive to tensile with increasing N₂ flow, in which an N₂ flow of 20 sccm provided an almost strain-free film. The compressively strained 12.6 nm thick film at 5 sccm N₂ resulted in lower ρ_{RT} (~139μΩcm) and superconducting critical temperature (T_c~12.26K). Measured T_c is similar or higher than reported T_c (12.10K) of 15nm thick ALD NbN films [3]. For an optimized 30nm thick film, carbon is below the XPS detection limit, RMS surface roughness is 0.52nm, and rocking curve FWHM is 0.69°, which is narrower than previously reported for 30 nm thick films [3]. T_c on all these films were also measured to establish process-structure-property relationships, and results will be discussed in the context of use in quantum and high temperature contact applications.

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11:45am AP+PS+TF-WeM-16 Thin Film Property Modification via Electric Field-Modulated Atomic Layer Deposition, Jessica Jones¹, Shi Li, Francisco Lagunas Vargas, Zachary Hood, Argonne National Laboratory

Thin, conformal film growth via atomic layer deposition (ALD) is broadly used in microelectronics, photovoltaics, and other industries. Enhanced thin film properties are required to advance device performance. Electric fields affect gas phase molecules, and adsorption behavior, but have not been extensively investigated for direction of thin properties. Static electric fields are generated and maintained *in situ* inside an ALD reactor resulting in modification of crystallinity and chemical composition. Thicknesses were determined via spectroscopic ellipsometry, uniformity was investigated by atomic force microscopy, crystallinity by x-ray diffraction and (scanning) transmission electron microscopy (S)TEM, and chemical composition by x-ray photoelectron spectroscopy. These systems are computationally investigated to probe the mechanism by which the ALD processes are enhanced.

Acknowledgements: This material 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. Work performed at the Center for Nanoscale Materials, a U.S. Department of Energy Office of Science User Facility, was supported by the U.S. DOE, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

Wednesday Morning, September 24, 2025

12:00pm **AP+PS+TF-WeM-17 Thin Conductive Cu Films by In-Situ Plasma Post-Reduction of Atomic Layer Deposited CuO**, *Maria Sales, Neeraj Nepal, Peter Litwin, David Boris, Scott Walton, Virginia Wheeler*, Naval Research Laboratory

Interconnect applications in microelectronics has helped spur the need to develop robust and scalable atomic layer deposition (ALD) processes for copper (Cu). For this application space, the unique advantage of ALD is being able to conformally coat via structures with high aspect ratios due to the self-saturating nature and precise thickness control. Reported ALD recipes for pure Cu typically rely on reactions between a metal-organic Cu precursor and either a thermal or plasma reducing reactant. However, these conventional ALD Cu processes have very low growth rates. Like other metal ALD recipes, ALD Cu typically requires thicknesses of at least 20-40 nm to achieve a fully coalesced, conductive film. Thus, limiting these process in applications where ultrathin highly conductive layers are required.

In this work, we report on an alternative way to obtain conductive Cu thin films by combining CuO with a higher growth rate and faster coalescence with an in-situ plasma reduction. Initially, copper (II) oxide, or CuO, is deposited by PEALD at a substrate temperature of 150 °C, using copper(I)-N,N'-di-sec-butylacetamidinate ([Cu('Bu-amd)]₂) and Ar/O₂ plasma as precursors. The growth rate for this CuO recipe is 0.3 Å/cycle, which is higher than what is obtained for pure Cu using the same precursor (0.1 Å/cycle). Grown CuO films have a low concentration of incorporated ligands and a smooth surface morphology. Following a fixed number of CuO ALD cycles, the CuO film is then exposed to in-situ reducing Ar/H₂ plasma pulses. To characterize the resulting films, spectroscopic ellipsometry (SE), X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), and contactless sheet resistance measurements were performed.

Various parameters during the Ar/H₂ reducing plasma, such as total exposure time, pulse lengths, and number of reducing plasma cycles, were investigated and effect on key properties of the resultant Cu film, such as chemistry, morphology, and resistivity will be discussed. Additionally, we report on utilizing supercycles of CuO ALD and reducing plasma pulses to grow thicker (30 nm) Cu films with low resistivity. To date, our most optimal CuO-then-post-reduction procedure yielded a 30 nm Cu film with a root mean square (RMS) roughness of 3.3-3.5 nm and a resistivity of 3.8 μΩ cm, which is only a factor of 2 greater than for bulk Cu.

Plasma Science and Technology

Room 201 ABCD W - Session PS-WeM

Plasma Catalysis and Surface Interactions

Moderator: Sumit Agarwal, Colorado School of Mines, USA

8:00am **PS-WeM-1 The Impact of Plasma Enhancement Gasses on Hydrogen Superpermeation Through Palladium Alloy Metal Foil Pumps**, *Caroline Hufnagel, Vitor Camacho*, Colorado School of Mines; *Thomas Fuerst*, Idaho National Laboratory; *Doug Way, Colin Wolden*, Colorado School of Mines

The fusion reaction between the hydrogen isotopes deuterium and tritium is the most likely path to enabling fusion energy. The efficient processing of the tritium is critical to the plant operation for fusion energy systems. Metal foil pumps (MFPs) are the leading technology for direct internal recycling (DIR) of hydrogen isotopes from the plasma exhaust in future fusion plants. MFPs rely on the concept of superpermeation, where H atoms generated by a plasma directly absorb into the metal foil, rapidly diffuse, and desorb downstream. To date, studies of superpermeation have predominantly employed pure hydrogen. In practice the plasma exhaust may contain significant levels of plasma enhancement gasses (PEGs, i.e. Ar, Ne, Kr). These inert gasses have metastable states that can enhance plasma density and stability. In this work, we systematically study the impact of PEG addition on the hydrogen superpermeation performance of PdCu and PdAg MFPs operated at low temperature (< 200 °C). In the case of argon, flux enhancements of up to 60% relative to pure H₂ plasma were observed, with the optimal concentration range being 5-10 % Ar. Performance correlated with the optical emission of the atomic H, and benefits were more appreciable on PdAg than PdCu. Beyond 15% addition plasma enhancement benefits were offset by dilution. Interestingly, the level of permeation enhancement improved with increasing DIR fraction, and potential reasons for this phenomenon are discussed.

Depending on programming this may be a better fit in PS8: Sustainability and Plasmas

8:15am **PS-WeM-2 The Impact of Contaminants on Superpermeation Through Palladium Alloy Metal Foil Pumps**, *Chao Li*, Marathon Fusion; *Caroline Hufnagel*, Colorado School of Mines; *Thomas Fuerst*, Idaho National Laboratory; *Vitor Camacho, Doug Way, Colin Wolden*, Colorado School of Mines

The fusion reaction between the hydrogen isotopes deuterium and tritium is the most likely path to enabling fusion energy. The efficient processing of the tritium is critical to the plant operation for fusion energy systems. Metal foil pumps (MFPs) are the leading technology for direct internal recycling (DIR) of hydrogen isotopes from the plasma exhaust in future fusion plants. MFPs rely on the concept of superpermeation, where H atoms generated by a plasma directly absorb into the metal foil, rapidly diffuse, and desorb downstream. To date, studies of superpermeation have predominantly employed pure hydrogen. Though undesired, inevitably MFPs will be exposed to unintentional contaminants such as carbon and oxygen, with consequences for long term durability. In this work, a combination of surface analytical techniques (Auger, XPS) and superpermeation experiments were employed to understand the impact of common contaminants. The critical nature of clean surfaces is first demonstrated through compression experiments using asymmetrically prepared PdCu MFPs. Contamination degrades performance, inhibiting both absorption of superthermal hydrogen on the feed surface and recombinative desorption on the permeate surface, with the latter being more detrimental. Next, sputter-cleaned MFPs were placed in a chamber that had been pre-contaminated with carbon. Upon plasma ignition, the superpermeation rate initially increased before rapidly declining to zero. The initial increase suggests that very small (submonolayer) contamination levels are perhaps beneficial, but would be difficult to control. O₂ plasma exposure could largely restore the performance of carbon-contaminated PdAg MFPs, but proved detrimental to PdCu. Surface analysis techniques are employed to understand the differences between the two alloys.

8:30am **PS-WeM-3 Elucidating Complex Interactions in Non-Thermal Plasma-Assisted Reactions on (Supported) Porous Catalysts**, *Michele Sarazen*, Princeton University

INVITED

Reactions such as dry reforming of methane (DRM) and ammonia synthesis are significant targets for renewable electricity driven manufacturing, given their current dependence on fossil fuels. Plasma-assisted catalysis, which aims to activate reactants near bulk ambient temperature and pressure, is one of these approaches. While plasma alone can activate these molecules, it is often unselective, requiring a catalyst to tailor the selectivity to desired products. Further, plasma-assisted catalysis can demonstrate synergistic effects due to the interactions of the plasma discharge on the catalyst and vice versa. However, effective catalyst design is limited by the lack of fundamental understanding of these plasma/catalyst interactions during reaction. In this work, we demonstrate the use of a dielectric barrier discharge (DBD) plasma, in part interfaced with a diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) reaction chamber, for ammonia synthesis, DRM, and related chemistries on various oxide supports (with metal nanoparticles). We elucidate impacts of surface species and porosity/functionality of porous oxide supports on plasma properties, catalytic activity, therefore the energy yield of DBD-assisted catalysis, which is required for implementation.

9:00am **PS-WeM-5 In Situ/Operando Diagnostics of Liquids and Catalysts in Contact with Plasmas**, *Kasidapa Polprasarn, Dihya Sadi*, Laboratoire de Physique des Plasmas (CNRS, Ecole Polytechnique, Institut Polytechnique de Paris, Sorbonne Université), France; *Darwin Kurniawan*, Department of Chemical Engineering, National Taiwan University of Science and Technology, Taiwan; *Thomas Orrière*, Institut PPRIME (CNRS, Université de Poitiers, ISAE-ENSMA), France; *Pankaj Pareek*, Faculty of Mathematics, Physics, and Informatics, Comenius University, Slovakia; *Francesca Caielli, Karthik Thyagarajan*, Institut PPRIME (CNRS, Université de Poitiers, ISAE-ENSMA), France; *Mario Janda*, Faculty of Mathematics, Physics, and Informatics, Comenius University, Slovakia; *Wei-Hung Chiang*, Department of Chemical Engineering, National Taiwan University of Science and Technology, Taiwan; *Olivier Guaitella, David Pai*, Laboratoire de Physique des Plasmas (CNRS, Ecole Polytechnique, Institut Polytechnique de Paris, Sorbonne Université), France

INVITED

Plasma interactions with liquids and solids are at the core of plasma electrochemistry (PEC) and plasma-assisted catalysis (PAC), respectively. To address the need for direct measurements in the plasma-liquid and plasma-

catalyst interfacial regions, respectively, we employ an *in situ/operando* approach using multiple diagnostic techniques to study a range of physical and chemical properties at plasma interfaces. The centerpiece of this platform is *in situ/operando* spontaneous Raman microspectroscopy. For a PEC batch reactor in pure water, this technique revealed that the concentrations of aqueous H_2O_2 and NO_3^- at a depth of a few tens of microns from the plasma-liquid interface are greater than in the bulk liquid [1]. We have also successfully performed *in situ/operando* Raman on an electrospray reactor [2]. Here, we will present two case studies of changes to Raman spectra observable only in the presence of plasma. First, for PEC in air plasma-water systems at atmospheric pressure, we will focus on the spectral profile of the $-\text{OH}$ stretch band of water and of probe molecules such as NO_3^- . Analysis of $-\text{OH}$ stretch indicates that the plasma disrupts the hydrogen bonding network of water. To assist in pinpointing the cause, we will examine the broadening of the N-O symmetric stretch mode (ν_1) of NO_3^- at less than 20 μm depth from the plasma-liquid interface. Second, we will present a study of a PAC reactor consisting of a low- to medium-pressure CO_2 plasma in contact with CeO_2 as a catalyst [3]. Tracking of the first- and second-order optical phonons of CeO_2 , as well as O-O bonds in superoxides, yields information on thermal effects and oxygen vacancy formation. Besides Raman spectroscopy, additional *in situ/operando* diagnostics have been employed when monitoring the synthesis of graphene quantum dots by PEC reactors [4]. We tracked their production via photoluminescence (PL) and UV-VIS absorption spectroscopies. Both the PL and absorption signals achieve their peak intensity not at the interface but at a depth of several millimeters. This aligns with liquid flow field measurements by particle image velocimetry, which indicate the presence of a low-velocity zone at this depth.

References

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9:30am **PS-WeM-7 Catalytic Deconstruction Product Tunability Through Atmospheric Air Plasma Pre-Treatment, Aunice Goodin¹**, North Carolina State University; *Tridip Das*, California Institute of Technology; *Shashwata Chakraborty*, *Suboy Bepari*, *Debasish Kuila*, North Carolina Agricultural and Technical State University; *William Goddard*, California Institute of Technology; *Steven Shannon*, North Carolina State University

The large amount of waste plastic produced is becoming a more prominent concern, as production of single use plastic continues to increase. Catalytic deconstruction is viewed as one potential method for deconstruction of waste plastics into usable material. In this work, we use an atmospheric pressure air plasma to pre-treat polypropylene, to better facilitate reaction with the catalyst. It has been found that this pre-treatment can tune the products of the catalytic deconstruction to change the distribution of products as needed.

Plasma treatment is performed in a sealed vial with a rubber septum cap. A steel hypodermic needle is used to both deliver synthetic air and apply a high voltage in a pin-to-cup configuration. The grounding cup is made up of a copper pipe soldered to a copper plate. The vial is placed the copper “cup” with the whole assembly surrounded with a 3D printed holder to prevent arcing from the high voltage to ground. A second hypodermic needle is then used to allow air to escape. Treatments range from 15-120 minutes at 30-40 kV (6-34 W), a frequency of 450 Hz, and a flow rate of 0-400 SCCM.

The catalyst used was a ZSM-5/SBA-15 composite catalyst, with the ZSM-5 added into the SBA-15 structure during its production. This was reacted with an equal ratio of catalyst to plastic at 400 °C with a constant flow of 1.5 SCCM of N_2 . This treatment was continued until products were no longer detected using gas chromatography mass spectrometry (GC/MS).

The best total conversion of 94% was observed with a 30 kV treatment for 30 minutes, at a flow rate of 110 SCCM, with a major product of propene (66%). The major product, as well as overall product distribution can be changed by performing the plasma treatment under different voltage, time, and flow rate.

Molecular dynamics simulations have shown the mechanism of the plastic degradation in plastic to be due to a reaction with ozone. Ozone measurements were taken to investigate different conditions. The plasma and product plastics will be further investigated through MALDI-TOF and OES measurements to better understand the conditions needed to produce specific products. The mechanism can then be better illuminated to understand how this could be applied to catalytic deconstructions with different scaled up or different plasma systems.

Through variations in plasma pre-treatment, catalytic deconstruction can be tuned to produce different product distributions. While this cannot shift the total distribution of products, it could increase the viability of catalytic deconstruction despite market fluctuations.

This material is based upon work supported by U.S. Department of Energy (DOE) no. DE-EE0009945.

11:00am **PS-WeM-13 Gas-Phase Plasma Synthesis as a Method for Producing Nanomaterials with Special Properties, Hartmut Wiggers**, University of Duisburg-Essen, Germany

INVITED

Gas phase synthesis has been an established process for the production of functional nanoparticles for decades. The manufacturing processes used are dominated by flame processes, whereby mostly oxide materials are produced. However, with the increasing use and storage of renewable energies for industrial processes, there is a growing need for new types of materials that can often no longer be produced using flame-based processes alone. Gas-phase plasma synthesis offers new possibilities here, as an essential prerequisite of flame processes – the use of a reactive gas mixture consisting of fuel and oxidizer – is not required, which opens up access to oxide-free materials in particular. At the same time, plasma processes can be operated over a wide pressure and temperature range, which can be used for setting specific temperature-time profiles.

Microwave and ICP plasma processes are particularly suitable for the production of high-purity materials in technically relevant quantities, as the coupling of energy into the gas phase process is contactless. Using the example of selected materials based on carbon (especially few-layer graphene, FLG) and silicon, it is shown how specific materials for applications in the field of energy conversion and storage as well as for catalysis can be produced using plasma processes. Starting from the first steps of nanoparticle formation, possibilities are shown to further develop the synthesis up to the production of functional composite materials as well as single-atom catalysts.

11:30am **PS-WeM-15 Interaction of Etching Plasmas with Polyurea Films deposited by Molecular Layer Deposition for Surface and Sidewall Passivation, Wallis Scholl**, Colorado School of Mines; *Thorsten Lill*, *Mingmei Wang*, *Wenyu Zhang*, *Harmeet Singh*, Lam Research Corporation; *Sumit Agarwal*, Colorado School of Mines

INVITED

Molecular layer deposition (MLD) is a vapor-phase process of alternating surface reactions which can be used to grow organic and hybrid organic-inorganic films. MLD films have several potential applications in semiconductor processing, including as conformal coatings or as a protective layer during plasma etching. However, the use of MLD in practical applications is hindered by the complexity of the growth mechanism. Some bifunctional molecules will react to the film surface with both of their functional groups, thereby consuming reactive sites and lowering the growth per cycle (GPC). Molecules can also be added to the film through physisorption to the growth surface, which adds new reactive sites to the film. Further, we have found that different MLD precursors can have different rates of double reaction and physisorption. Molecule chain length, flexibility, and hydrogen bonding must be carefully considered when selecting an MLD chemistry, as they all affect the film growth. The physisorption contribution to film growth can also be promoted by lowering the deposition temperature, which results in an increase in GPC. However, films with a high degree of physisorbed material can be unstable, as this weakly physisorbed material can later diffuse out.

In this work, toluene diisocyanate (TDIC) and ethylene diamine (ED) were used as precursors for MLD of polyurea, which was grown on top of a SiO_2 starting surface. The films were then exposed to a HF plasma to evaluate their interactions with reactive plasma. During initial HF exposure, only the MLD film was etched, while the underlying SiO_2 was protected. As plasma exposure continued, eventually removal of the MLD film stopped and the SiO_2 was selectively etched. The length of the SiO_2 etch delay was found to be highly dependent on the morphology of the MLD film; films with a higher degree of interconnectivity were better able to prevent F from diffusing through the film to access the SiO_2 . For example, depositing at a higher deposition temperature, which reduces physisorption into the film,

Wednesday Morning, September 24, 2025

resulted in a higher etch resistance. Ion bombardment was also found to increase film interconnectivity, thereby preventing SiO₂ etch. Additionally, we deposited MLD films on high aspect-ratio (HAR) trenches to evaluate the film conformality. While the analogous process of atomic layer deposition (ALD) requires very high doses to provide necessary diffusive flow into the trench, we found that during MLD of polyurea, films are deposited conformally using the saturation doses for a flat surface. We've attributed this to film reconstruction during MLD, which includes migration of physisorbed molecules through the film.

12:00pm **PS-WeM-17 Characterization and Operation of a 2-D Plasma Reactor for Methane Pyrolysis**, *Huseyin Ozturk, Andac Yagiz Kaya, Necip Berker Uner*, Middle East Technical University, Turkey

Methane pyrolysis is breaking the C-H bonds of methane in an O₂-free atmosphere. This process can synthesize H₂ directly from natural resources along with solid carbon with no carbon dioxide emissions. Using an atmospheric plasma for methane pyrolysis is attractive since the process is fully electrified, is completely intermittent with very rapid turn-on/off times and requires no consumables. However, the number of plasma reactors demonstrated for methane pyrolysis is very limited, and the application of chemical reaction engineering fundamentals to the design and operation of these reactors is currently absent in literature.

This talk focuses on the design and characterization of a unique, 2-dimensional, gliding arc (GA) plasma reactor for conducting methane pyrolysis at atmospheric pressure to produce carbon black and hydrogen. The GA is a *warm* plasma, and it combines high-temperature thermal conversion with non-equilibrium electron-impact chemistry. The reactor flow was confined to be unidirectional such that nearly all the gas was put in contact with the GA, thereby increasing flow utilization in the reactor, as verified by computational fluid dynamics. This resulted in improvements on the experimentally measured residence time distribution, and GA formation was visually and electrically analyzed in the confined flow geometry under direct-current excitation. Temperature measurements and heat transfer modeling indicated that the thermal efficiency of the reactor can be made to be as high as 50%, meaning that half of the electrical energy was converted to heating the gas, whereas the rest was dissipated into the ambient through the reactor body. This presentation will elaborate on CH₄ conversion, as well as continuous collection and ex-situ characterization of the carbon product, primarily through Raman and FTIR spectroscopy. The effect of using argon in the mixture and the influence on reactor materials to CH₄ conversion and H₂ selectivity will also be presented.

Atomic Scale Processing Mini-Symposium Room 206 A W - Session AP+PS+TF-WeA

Thermal and Plasma enhanced Atomic Layer Etching

Moderators: Eric Joseph, IBM T.J. Watson Research Center, Greg Parsons, North Carolina State University

2:15pm AP+PS+TF-WeA-1 Selectivity During Spontaneous Dry Thermal Etching of Si-Based Materials by Hydrogen Fluoride, Marcel Junige, Micah Duffield, Steven George, University of Colorado at Boulder

Spontaneous dry thermal etching involves reaction of a thin film surface with a gaseous etchant leading to material removal with a constant etch rate. Spontaneous dry thermal etching can often be involved as a competitive process during thermal atomic layer etching (ALE). Selectivity can occur during spontaneous dry thermal etching with hydrogen fluoride (HF) because HF can form different etch species, F^- or HF_2^- , that etch with material specificity. For example, F^- can etch SiN_x and HF_2^- can etch SiO_2 . The nature of the active HF etch species can be controlled by the HF environment. HF alone yields F^- etch species. HF together with a polar co-adsorbate can yield HF_2^- etch species.

The talk will discuss four examples of HF selectivity: SiN_x etch vs SiO_2 non-etch; Si etch vs Si_3N_4 , $SiCOH$ and SiO_2 non-etch; Si etch vs Si non-etch with co-adsorbed H_2O ; and SiO_2 non-etch vs SiO_2 etch with co-adsorbed NH_3 or $(CH_3)_2NH$ (dimethylamine). The experiments were conducted using *in situ* spectroscopic ellipsometry to monitor the film thicknesses during time to obtain etch rates. Additional quadrupole mass spectrometry (QMS) analysis was able to monitor the presence or absence of etch products during the experiments for Si etch vs Si non-etch with co-adsorbed H_2O .

Selective SiN_x etch vs SiO_2 non-etch was observed for HF etching at 275 °C. Etch selectivity was measured for HF pressures from 0.5 to 9.0 Torr. SiN_x : SiO_2 etch selectivity approached a maximum of 150 : 1 at 9.0 Torr. These results are consistent with F^- as the active etch species that yields SiN_x etching. Si etch vs Si_3N_4 , $SiCOH$ and SiO_2 non-etch was also demonstrated for HF etching at 275 °C and an HF pressure of 3 Torr. Crystalline Si etched at 23 Å/min. In comparison, Si_3N_4 , $SiCOH$ and SiO_2 etched at much smaller rates of 0.03, 0.11 and 0.01 Å/min, respectively. Much higher Si etch rates were observed at higher HF pressures at 275 °C. The Si etch rate increased to 240 Å/min at an HF pressure of 9 Torr.

Si etch vs Si non-etch with co-adsorbed H_2O illustrated the influence of polar co-adsorbed species on the etching. QMS experiments revealed that Si was etched by HF at a pressure of 1 Torr with a temperature threshold at ~150 °C. In contrast, co-dosing H_2O at a pressure of 1 Torr eliminated Si etching. These results suggest that F^- is the active etch species for Si etching. QMS experiments also identified the volatile etch products as H_2 and SiF_4 . SiO_2 non-etch vs SiO_2 etch with co-adsorbed NH_3 or $(CH_3)_2NH$ also supported the idea that polar co-adsorbates convert the HF active species to HF_2^- . Without polar co-adsorbates, F^- species do not etch SiO_2 . With polar co-adsorbates, HF_2^- species can etch SiO_2 .

2:30pm AP+PS+TF-WeA-2 ZrO₂ Thermal Atomic Layer Etching Using HF for Fluorination and TiCl₄ for Ligand Exchange: Effect of Processing Parameters, Chen Li, Troy Collieran, University of Colorado Boulder; Beomseok Kim, Hanjin Lim, Samsung Electronics Co., Republic of Korea; Steven George, University of Colorado Boulder

ZrO₂ thermal atomic layer etching (ALE) can be performed using sequential surface modification and volatile release reactions. HF fluorinates the ZrO₂ surface to form a ZrF₄ layer. TiCl₄ then undergoes ligand-exchange and volatilizes the ZrF₄ layer. In this study, the etch rate of ZrO₂ ALE was evaluated as a function of various processing parameters such as pressure, temperature and exposure time. The initial ZrO₂ films were grown by atomic layer deposition (ALD) using tetrakis(diethylamino) zirconium and H₂O. The processing parameters during ZrO₂ thermal ALE were examined using various techniques including quartz crystal microbalance (QCM), x-ray reflectivity (XRR), atomic force microscopy (AFM) and quadrupole mass spectrometry (QMS). *In situ* QCM experiments examined ZrO₂ ALE at HF pressures from 0.1 to 0.9 Torr with fixed TiCl₄ pressure and at TiCl₄ pressures from 0.2 to 2 Torr with fixed HF pressure. The mass of the ZrO₂ film decreased linearly with number of ALE cycles. The higher HF and TiCl₄ pressures led to higher ZrO₂ etch rates. However, self-limiting behavior was observed at both low and high HF and TiCl₄ pressures. The ZrO₂ etching rates were also observed to increase at higher temperatures. These results illustrate that self-limiting reactions can occur over a range of reactant pressures and temperatures. At higher reactant pressures, the QCM

analysis measured mass change per cycle (MCPC) values that varied from ~-49.4 to ~-118.6 ng/(cm² cycle) at 200 and 300 °C, respectively. These MCPCs correspond to ZrO₂ etch rates from 0.87 to 2.09 Å/cycle at 200 and 300 °C, respectively. XRR measurements also confirmed the linear removal of ZrO₂ versus number of ALE cycles and the etch rates. AFM measurements also studied the roughness of crystalline ZrO₂ films after ALE. These crystalline films contained a mixture of monoclinic and tetragonal phases. The surface roughness increased with number of ALE cycles. However, higher precursor pressures at high temperatures produced a lower roughness increase. In addition, QMS analysis revealed the volatile etch products during the sequential HF and TiCl₄ exposures on ZrO₂ at 200, 250 and 300 °C. The signal intensity of the etch products increased at higher temperatures. H₂O was monitored during the HF exposure when HF fluorinates ZrO₂ to produce ZrF₄. ZrCl₄ was observed as the etch product and TiFCl₃ was detected as the ligand-exchange product during the TiCl₄ exposure. These products confirm the ligand-exchange reaction between TiCl₄ and ZrF₄. This project was supported by Samsung Electronics Co., Ltd (I0230707-06660-01).

2:45pm AP+PS+TF-WeA-3 SiO₂ Etching by HF in a Liquid-Like H₂O Layer in a Vacuum Environment, Samantha Rau¹, Micah Duffield, University of Colorado at Boulder; Antonio Rotondaro, Hanna Paddubrouskaya, Kate Abel, Tokyo Electron America, Inc.; Steven George, University of Colorado at Boulder

Adsorbed H₂O layers may be employed for etching by a liquid layer in a vacuum environment. Liquid-like H₂O layers can form at H₂O pressures around 10 Torr and temperatures around room temperature. Etchants may then be dissolved in the liquid-like H₂O layers. These conditions allow many etching processes that are conducted in wet aqueous solutions to be extended to liquid-like H₂O layers in vacuum.

This study focused on SiO₂ etching by HF in a liquid-like H₂O layer in vacuum. The experiments were conducted in a warm-wall vacuum chamber designed with a sample stage that allowed for H₂O liquid layer formation only on the cooled stage. The thickness of SiO₂ films was measured using *in situ* spectroscopic ellipsometry as the SiO₂ films were exposed to various H₂O and HF pressures at different substrate temperatures. Studies were conducted at H₂O pressures from 5 to 30 Torr, HF pressures from 2 to 6 Torr, exposures time from 2 to 20 s, and temperatures from 18.1 to 30.4 °C. The SiO₂ films etched readily under these conditions.

The SiO₂ etch rate increased versus HF pressure. Figure 1 shows that as the HF pressure was increased from 2 to 6 Torr, at 30.4 °C with a H₂O pressure of 15 Torr and exposure time of 5 s, the SiO₂ etch rate increased from ~14 Å/exposure to ~3315 Å/exposure, respectively. The SiO₂ etching also increased versus H₂O pressure. Figure 2 shows that as the H₂O pressure was increased from 10 to 30 Torr, at 30.4 °C with a HF pressure of 3.5 Torr and exposure time of 5 s, the SiO₂ etch rate increased from ~10 Å/exposure to ~105 Å/exposure, respectively. The dramatic variation in SiO₂ etch rates suggests that the thickness and composition of the liquid-like layer may be changing rapidly with HF and H₂O pressure.

The SiO₂ etch rate also increased versus exposure time. As the exposure time increased from 2 to 20 s, at 30.4 °C with a H₂O pressure of 10 Torr and HF pressure of 3.5 Torr, the SiO₂ etch rate increased from ~6 Å/exposure to ~150 Å/exposure, respectively. The SiO₂ etch rate was also inversely dependent on sample temperature. Experiments were conducted at temperatures of 30.4 °C, 27.2 °C, and 18.1 °C with a H₂O pressure of 15 Torr, HF pressure of 3.5 Torr, and exposure time of 5 s. These studies yielded SiO₂ etch rates of ~33 Å/exposure, ~1564 Å/exposure, and ~3456 Å/exposure, respectively. The large increase of the SiO₂ etch rate is attributed to the thicker liquid-like layer at lower temperatures. The thicker liquid-like layer may be able to more easily solvate the HF reactants and SiO₂ etch products.

3:00pm AP+PS+TF-WeA-4 Wet-Like Atomic Layer Etching of WCN by Applying the Leidenfrost Effect to Obtain Floating Nanomist-Assisted Vapor Etching, Thi-Thuy-Nga Nguyen, Nagoya University, Japan; Kazunori Shinoda, Kenji Maeda, Kenetsu Yokogawa, Masaru Izawa, Hitachi High-Tech Corp., Japan; Kenji Ishikawa, Masaru Hori, Nagoya University, Japan

Semiconductor devices have been miniaturized to the nanometer scale. Work function metals, made from various metals like TiAlC, TiC, TiN, and WCN, are used in field effect transistor gate stacks. Precise control of isotropic and selective atomic layer etching (ALE) of thin metal gate materials in 3D nanostructures is crucial for the next-generation logic semiconductor devices. This requires minimizing damage from sputter effects in plasma ALE, high temperatures in thermal ALE, and pattern

collapse in wet ALE. In our previous study, we developed a wet-like plasma etching method for a ternary metal carbide TiAlC [1]. This technique combines the advantages of wet etching (high isotropy and selectivity) and dry etching (high controllability). By using high-density vapor plasma at medium pressures, we generated a rich radical source of reactive species to significantly increase the reaction rate with the sample surface. This opens an avenue for developing our new dry ALE method, named wet-like ALE.

Here we have demonstrated the wet-like ALE for WCN material by sequentially exposing it to a rich radical source of O₂ plasma for surface oxidation at a relatively low temperature of less than 40 °C and removal of the modified layer (WO₃) by dissolving it in a highly volatile nanomist flow. The proposed nanomist phase is a mist-vapor phase with properties between the mist liquid and vapor phases, maintaining the wet properties of the liquid phase at a minimal mist size for nanodevice applications. At the Leidenfrost point, the nanomist floats on its own stable vapor cushion film over the whole sample surface [2]. By using the Leidenfrost effect, the modified layer can be dissolved in a stable vapor film existing under the floating nanomist or in a floating nanomist-assisted vapor. The nanomists were generated from liquids by our originally developed non-contact atomizer at room temperature. The high removal rate of the modified layer (WO₃) was obtained at a temperature higher than 130 °C that is considered as the Leidenfrost point of the nanomist produced from the aqueous liquid mixture, in which the WCN surface is supposed to be etched by the floating nanomist-assisted vapor at medium pressures. Self-limiting oxidation and removal of WCN by nanomist were achieved in both steps of the wet-like ALE cycle.

Acknowledgement

We would like to thank Dr. Yoshihide Yamaguchi (Hitachi, Ltd., Japan) and Mr. KuangDa Sun (Nagoya University) for the previous discussions about Leidenfrost effect and mist generation, respectively.

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3:15pm AP+PS+TF-WeA-5 Thermal Atomic Layer Etching of Hafnium–Zirconium Oxide (HZO) Using Organofluorides for Fluorination, Aziz Abdulagatov, Jonathan Partridge, University of Colorado at Boulder; Matthew Surman, ASM Microchemistry Ltd., Finland; Steven George, University of Colorado at Boulder

Thermal atomic layer etching (ALE) of various materials has previously been achieved using sequential fluorination and ligand exchange reactions where HF has been used as the fluorination source. In this work, organofluorides were employed as an alternative to HF. The thermal ALE of Hf_{0.5}Zr_{0.5}O₂ (HZO) was demonstrated using various organofluorides. The organofluorides were N,N-Diethyl-1,1,2,3,3,3-hexafluoropropylamine (Ishikawa's reagent (IR)), 1,1,2,2-tetrafluoroethyl dimethylamine (TFEDMA) and diethylaminosulfur trifluoride (DAST). IR, TFEDMA and DAST are common deoxyfluorination reagents.

HZO ALE was demonstrated using organofluoride exposure in combination with ozone (O₃) and boron trichloride (BCl₃) exposures. Ozone was used to remove carbon residue resulting from organofluoride adsorption. BCl₃ was employed for ligand exchange with the fluorinated surface to form volatile Hf and Zr chlorides and BCl_xF_y products. BCl₃ can also undergo conversion with HZO.

In situ spectroscopic ellipsometry (SE) observed the linear decrease of HZO film thickness. Under similar reaction conditions at 270 °C, crystalline HZO films with a thickness of 10 nm displayed etch rates of 0.1, 0.2, and 0.5 Å/cycle, using IR, TFEDMA, and DAST, respectively. Etching amorphous HZO using IR yielded higher etch rates of 0.6 Å/cycle at 270 °C. The IR, O₃ and BCl₃ surface reactions were also determined to be self-limiting.

Quadrupole mass spectrometry (QMS) was also utilized to study the IR-O₃-BCl₃ etch process on crystalline ZrO₂ powder at 270°C. During IR exposure, organic fragments and HF were detected indicating that HF is produced *in situ* by IR at 270 °C. During O₃ exposure, combustion products were observed from the oxidation of organic residuals left from IR exposures. During BCl₃ exposure, Hf and Zr chloride products, as well as BCl_xF_y products, were produced by the ligand-exchange reactions. Concurrently, boroxine ring (B₃O₃Cl₃) fragments were monitored and indicated the conversion of HZO to B₂O₃.

3:30pm AP+PS+TF-WeA-6 Selective Atomic Layer Etching of SiO₂ over Si₃N₄ via TMA Surface modification and SF₆ Remote Plasma, Jieun Kim, Min Kyun Sohn, Sun Kyu Jung, Min-A Park, Jin Ha Kim, Jaeseoung Park, Subin Heo, Sang-Hoon Kim, Jeong Woo Park, Seong Hyun Lee, Dongwoo Suh, Electronics and Telecommunications Research Institute, Republic of Korea

Precise etch selectivity between SiO₂ and Si₃N₄ is critical in advanced semiconductor fabrication processes, especially for applications such as spacer patterning in Gate-All-Around Field-Effect Transistors (GAAFETs) and multilayer structuring in 3D NAND devices. While selective etching of Si₃N₄ over SiO₂ has been widely studied using plasma chemistries such as SF₆/H₂/Ar/He, NF₃/O₂, and CF₄/O₂/N₂ gas mixtures,¹⁻³ achieving atomic-scale precision in the reverse case—preferentially etching SiO₂ over Si₃N₄—remains challenging.

In this work, we present an atomic layer etching (ALE) approach that enables highly selective etching of SiO₂ over Si₃N₄ through surface chemical engineering. The process sequence comprises four steps—trimethylaluminum (TMA) surface modification, Ar purge, SF₆ remote plasma exposure, and Ar purge—performed at 300 °C, 5 Torr, with an SF₆ flow rate of 50 sccm.

Under standard SF₆ plasma conditions, Si₃N₄ is typically etched more rapidly than SiO₂ due to the greater susceptibility of Si–N bonds to fluorine radicals. However, we found that incorporating a TMA surface modification step effectively inverts this trend. Chemical interactions at the surface are believed to yield Al–O–Si linkages on SiO₂ and Al–N–Si on Si₃N₄, leading to distinct reactivities during subsequent F-radical exposure. The Al–O–Si sites promote the formation of volatile AlF₃ and SiF₄, whereas Al–N–Si structures exhibit much lower fluorine reactivity.

This chemistry-driven mechanism enabled a marked difference in etch per cycle (EPC), with SiO₂ reaching 0.49 Å/cycle and Si₃N₄ reaching 0.05 Å/cycle, resulting in a selectivity close to 10:1. Whereas conventional atomic layer plasma etching using CH₂F₂/O₂/N₂ gas mixtures achieves selectivity through physical passivation or polymer deposition—often leading to surface damage or limited thickness control—our method, based on surface chemical modification and remote plasma exposure, enables damage-free etching and precise, layer-by-layer thickness control by decoupling chemical reactivity from ion bombardment. These findings suggest that surface modification-based selectivity tuning can serve as a viable strategy for precision etching in next-generation logic and memory device integration.

Reference

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4:15pm AP+PS+TF-WeA-9 Mitigating Redeposition in Directional Atomic Layer Etching of Lithium Niobate Using HBr Plasma, Ivy Chen, Caltech; Frank Greer, Jet Propulsion Laboratory (NASA/JPL); Austin Minnich, Caltech
Lithium niobate (LiNbO₃, LN) is a ferroelectric crystal of interest for integrated photonics owing to its large second-order optical nonlinearity and the ability to impart periodic poling via an external electric field. However, on-chip device performance based on thin-film lithium niobate (TFLN) is presently limited by propagation losses arising from surface roughness and corrugations. Atomic layer etching (ALE) could potentially smooth these features and thereby increase photonic performance. Previously, our group has reported the first isotropic ALE processes for lithium niobate. Here, we report a directional ALE process for x-cut MgO-doped LN using an HBr-containing plasma. At 0 degrees Celsius we report an 85% synergy ALE recipe with etch rate of 1.1 nm/cycle and surface roughening. At 200 degrees Celsius we report a reduced synergy at 30%, with an etch rate of 1.24 nm/cycle and no evidence of surface roughening. We also compare the surface roughness result of the HBr containing process with a chlorine-only process. Our ALE process could be to fabricate waveguide structures with nanometer precision without surface roughening or redeposition, thereby increasing the performance of TFLN nanophotonic devices and enabling new integrated photonic device capabilities.

Wednesday Afternoon, September 24, 2025

4:30pm AP+PS+TF-WeA-10 Atomic Layer Etching of Sputter-Deposited AlN Thin Films in Cl₂-Ar Plasmas, *Iurii Nesterenko*¹, Silicon Austria Labs GmbH, Austria; *Jon Farr*, Applied Materials, Inc.; *Steffen Harzenetter*, Applied Materials, Inc., Germany; *Dmytro Solonenko*, Benjamin Kalas, Thang Dao, Silicon Austria Labs GmbH, Austria; *Julian Schulze*, Ruhr University Bochum, Germany; *Nikolai Andrianov*, Silicon Austria Labs GmbH, Austria

Aluminum nitride (AlN) is a widely used material in micro- and nanoelectronics, particularly in photonics and MEMS devices. However, one of the critical challenges in the fabrication of AlN-based devices is achieving precise nanoscale etching while maintaining smooth surfaces and well-defined etch profiles. Atomic Layer Etching (ALE) is a promising approach the above-mentioned problems, which are particularly crucial in photonic applications, where surface roughness and deviations in profile angles can result in optical losses and inefficient mode confinement.

This study investigates the ALE of AlN thin films deposited via sputter deposition on an 8-inch wafer. The wafer was diced into 2 × 2 cm coupons, which were then attached to a SiO₂ thermal oxide carrier wafer. The experiments were performed in an Applied Materials™ Centura™ DTM Chamber using Cl₂ and Ar gases for the modification (Cl step) and ion bombardment (Ar step) steps, respectively. The thickness of the AlN thin films was measured via spectroscopic ellipsometry (Semilab SE-2000). Also, the ion energy distribution function (IEDF) was analyzed using an ion energy analyzer (Impedance Quantum).

The feasibility of ALE for sputter-deposited AlN thin films was successfully demonstrated. The etch per cycle (EPC) was found to be approximately a single monolayer of the wurtzite AlN crystal structure (Fig.1). The ALE energy window was determined by analyzing the IEDFs in the Ar step, revealing the energy range of around 75 eV, which is consistent with the previously reported data in the literature [1]. Furthermore, measurements of the AlN sputtering threshold under Ar bombardment (Fig. 1) indicated minimal sputtering contributions, suggesting that the synergy of the process could approach 100%. Further investigations will be conducted to quantify this synergy more accurately. Moreover, it was determined that the process is linear, e.g. the EPC is constant against the number of cycles (Fig.2). The AlN RMS roughness after processing within the ALE energy window is around 570pm (Fig.3), which is lower than the original material roughness of 3nm.

Future work will also focus on optimizing the process by minimizing the duration of the Ar, Cl, and purge steps. The optimization of the Cl step will be complemented by X-ray Photoelectron Spectroscopy (XPS) to gain deeper insights into the surface chlorination mechanism.

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4:45pm AP+PS+TF-WeA-11 Sub-Surface TiO₂ Atomic Layer Etching (ALE) Through W Films, *Hannah Margavio*, Gregory Parsons, North Carolina State University

The growing complexity of microelectronic architectures requires the development of novel atomic-scale fabrication techniques. Traditional semiconductor processing relies on separate deposition and etching steps. For example, a common fabrication technique known as etch-replacement deposition proceeds with W atomic layer deposition (ALD) and TiO₂ chemical vapor etching (CVE) occurring locally via SiH₄ and WF₆ exposure, yielding a W film thickness roughly equal to the removed TiO₂ film. In the etch replacement process, WF₆ converts TiO₂ into an intermediate solid phase, TiWO_xF_y, which becomes volatile upon further WF₆ exposure. Alternatively, TiWO_xF_y can be reduced by SiH₄, resulting in a W-rich film.

In this work, we address the need for advanced and unique processing for more complex metal nanostructures using atomic layer etching (ALE). We demonstrate the fabrication of intricate metal architectures via sub-surface etching of TiO₂ by controlling WF₆, MoF₆, and BCl₃ etching conditions after W ALD. First, 30 W ALD cycles were deposited on TiO₂/Si line patterns resulting in ~20 nm of W deposition on TiO₂. Following deposition, the film stack was exposed to 10, 80, and 150 WF₆ individual doses. After WF₆ exposure, it was found the W layer remained and the underlying TiO₂ layer was etched away as a function of CVE cycles, creating an air gap between the patterned TiO₂ lines and the W layer. We will show when additional WF₆

doses were exposed to the film stack, the air gap spacing increased. Similarly, MoF₆ doses after W ALD initiated sub-surface TiO₂ CVE. With MoF₆, we were able to elucidate the sub-surface etching mechanism via STEM EDS mapping; we observed metal fluoride diffused through the W film to react with the underlying TiO₂, while etch products diffused out. Compared to WF₆ and MoF₆ driven CVE, ALE using sequential WF₆ and BCl₃ doses accelerated etching and allowed greater control of TiO₂ removal. By integrating W ALD and TiO₂ ALE with sequential WF₆ and BCl₃ cycles on patterned TiO₂ structures, unique film stacks with tunable, uniform air gaps were fabricated.

5:00pm AP+PS+TF-WeA-12 Pulsed Plasma Strategies for High-Precision Pseudo-Atomic Layer Etching, *Maryam Khaji*, University of Michigan; *Qinzhao Hao*, Mahmoud A. I. Elgarhy, Jeremy Mettler, University of Houston; *Hyunjae Lee*, Sang Ki Nam, Mechatronics Research, Samsung Electronics Co, Republic of Korea; *Vincent Donnelly*, University of Houston; *Mark J. Kushner*, University of Michigan

Conventional plasma-based atomic layer etching (ALE) involves two self-limiting steps: passivation, where radicals (e.g., Cl) passivate the top layer of the substrate (e.g., silicon) to form SiCl_x; and etching, where the passivated layer is selectively removed by an ion-rich flux with its energy tuned to etch only the passivated material [1]. In spite of its high precision, ALE is time-consuming due to the need to evacuate the chamber between steps and so is challenged to incorporate into high volume manufacturing (HVM). Strategies are needed to maintain the precision of ALE while increasing its processing speed.

In this work, we report on a computational investigation of strategies to achieve rapid and precise Pseudo-Atomic Layer Etching (P-ALE) processes. This investigation is conducted for an inductively coupled plasma (ICP) reactor with RF or dc power applied to the substrate using Ar/Cl₂ mixtures for Si etching. Reactor scale plasma properties are addressed using the Hybrid Plasma Equipment Model (HPEM). Feature profile evolution is evaluated using the Monte Carlo Feature Profile Model (MCFPM) [2].

We will discuss strategies for P-ALE whose goal is to maintain the dual-process (passivation-etching) of conventional ALE while using a single gas mixture. These strategies use combinations of pulsed source (ICP) and bias powers, and electrode biasing, that produce a passivation phase where ion energies are low, and that appears to be ion starved; followed by rapid etch phase where additional passivation is low, and that appears to be neutral starved. To achieve these ends, plasma potential and dc bias must be carefully managed. Comparisons are made to experimental data.

This work was supported by Samsung Electronics and the Department of Energy Office of Fusion Energy Sciences.

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5:15pm AP+PS+TF-WeA-13 Development of Atomic Layer Etching Process Dedicated to Diamond Electronic Devices, *Marine Régnier*, Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel; Institute of Applied Physics, University of Tsukuba; Japanese-French Laboratory for Semiconductor Physics and Technology J-FAST, CNRS, Univ. Grenoble Alpes, University of Tsukuba, France; *Aboulaye Traoré*, LSPM, CNRS, Université Sorbonne Paris Nord, France; *Marceline Bonvalot*, Univ. Grenoble Alpes, CNRS, Grenoble INP, LTM; Japanese-French Laboratory for Semiconductor Physics and Technology J-FAST, CNRS, Univ. Grenoble Alpes, University of Tsukuba, France; *Etienne Gheeraert*, Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel; Institute of Applied Physics, University of Tsukuba; Japanese-French Laboratory for Semiconductor Physics and Technology J-FAST, CNRS, Univ. Grenoble Alpes, University of Tsukuba, France

Diamond power devices, such as Schottky diodes and MOSFETs are currently being intensively investigated for possible application in power electronics and require dedicated fabrication processes to achieve adequate operating performances. Conventional etching techniques often lead to defects, surface roughness and sub-surface damages, which can significantly degrade carrier mobility and breakdown voltage of power devices. Thus, it becomes essential to develop diamond etching processes minimizing induced defects. Atomic layer etching (ALE) is a very soft etching technique involving two successive self-limiting and independent reactions. The first self-limiting reaction involves modifying the surface of a material by forming an ultra-thin reactive surface layer, while the second self-limiting reaction consists in the sputtering of the modified layer while keeping the underlayer intact. The repetition of these two reactions allows the removal of a layer of materials with a defect-free etched surfaces and sub-surfaces at atomic-scale precision. The first report of ALE of diamond

dates back to 1988 [1], however, since then, no further studies have been reported.

In this work, the ALE process optimization of (100) diamond is presented. The ALE process is achieved by first modifying the surface and then using a soft plasma to induce the selective removal of this modified surface. Experiments have been performed in a standard inductively coupled plasma reactive ion etching equipment with in-situ plasma monitoring by optical emission spectroscopy. They have been characterized as a function of the etching rate per cycle (EPC) estimated from diamond etched depth after 100 ALE cycles. The impact of the incident ionic bombardment kinetic energy during the 2nd ALE reaction has been evaluated from the dc self-bias voltage (V_{oc}). Results show a clear plateau of approximately 5 V (Fig. 1), called ALE window, demonstrating the self-limiting effect of the etching process within one ALE cycle. The etching rate is of 7.1 Å per cycle, corresponding to the removal of two (100) diamond monolayers per cycle. Finally, synergy factor has been calculated. Synergy measures the effect of combining the two ALE steps. Separately, 100 cycles of step 1 then 100 cycles of step 2 leads to an etching rate of 4.0 Å per cycle. But 100 cycles of (1+2) steps lead to 7.1 Å per cycle, *i.e.* a synergy of 43%. This again demonstrate the effectiveness of the ALE process.

All these results will be presented in detail and discussed in the light of literature data.

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5:30pm **AP+PS+TF-WeA-14 Atomic Layer Etching for Vertical Trench Control and Electrical Optimization in HDLK Materials**, *Sanghyun Lee, Keun Hee Bai*, Samsung Electronics, Republic of Korea

As device scaling continues, it becomes increasingly challenging to enhance device performance. In order to improve device performance, reducing resistance and capacitance in the BEOL (Back-End of Line) is especially important. Among various methods, minimizing damage to low-k dielectric materials during patterning processes has become a key challenge in BEOL integration. In this work, we suggest using Atomic Layer Etching (ALE) to overcome this problem, along with the selection of suitable low-k materials. ALE enhances controllability over surface reactions and profile formation by utilizing low ion energy, which enables the achievement of vertical profiles while simultaneously minimizing Plasma-Induced Damage (PID). The proposed ALE process utilizes a fluorocarbon-based surface modification step (C_4F_8), followed by a low-energy O_2 plasma step for selective carbon removal. This cyclic approach enables atomic-scale material removal with minimal physical damage, significantly reducing ion bombardment effects. To evaluate the effect of ALE on different low-k materials, we tested various High-Density Low-k (HDLK) samples with differences in k-value, modulus, and carbon composition. As a result, both low-k damage and vertical trench profile integrity were substantially improved, with smoother sidewalls and better verticality observed. The process performance was evaluated through detailed compositional analysis (XPS, EDX), PID characterization, and electrical measurements. The results confirmed that the proposed ALE method effectively reduced damage to low-k materials while enhancing profile control. Consequently, it demonstrates strong potential as a next-generation patterning solution for advanced BEOL integration.

5:45pm **AP+PS+TF-WeA-15 Mechanisms of Atomic Layer Etching of Ni₃Al**, *Owen Watkins, Taylor G. Smith*, University of California, Los Angeles; *Jean-François de Marneffe*, IMEC, Belgium; *Jane P. Chang*, University of California, Los Angeles

New metals and alloys are being investigated as potential replacements to TaBN in the absorber layer of extreme ultraviolet (EUV) lithography masks. Among potential candidates, Ni₃Al is particularly promising because it has both a high extinction coefficient and an index of refraction close to 1. A major hurdle in integration is anisotropically etching Ni₃Al selective to Ru, the 2-3 nm capping layer underneath the Ni₃Al absorber, with previously developed reactive ion etch and oxygen plasma-based atomic layer etch (ALE) having selectivities of 0.4 and 0.6, respectively. Better selectivity could be obtained through an ALE process based on cycles of nitrogen plasma, which does not spontaneously form volatile Ru compounds.

In this work, a Ni₃Al ALE process using nitrogen plasma, formic acid vapor, and Ar⁺ ion beam sputtering is investigated. The three step ALE process was shown to etch blanket Ni₃Al films at a rate of 1.0 nm/cycle. The self-limiting nature of the ALE process was examined by varying the duration of the nitridation, FA vapor, and Ar⁺ ion beam steps one at a time and measuring the resulting etch rate per cycle after 10 ALE cycles. These experiments

showed that increasing the low energy Ar⁺ ion beam served only to remove residual formate from the surface prior to starting the subsequent ALE cycle and was not responsible for etching the Ni₃Al. The anisotropy of the Ni₃Al ALE process was examined using specially prepared samples of Ni₃Al deposited over patterned Si which had an initial sidewall Ni₃Al thickness of 17 nm. Scanning electron microscopy (SEM) showed that 30 ALE cycles redeposited material on the feature sidewalls, increasing the sidewall thickness to 34 nm at the bottom of the patterned feature and 21 nm near the top. The etch mechanism, particularly the volatile Al product, was investigated by comparing the etch rates of Ni, Ni₃Al, NiAl, and Al films. Ni etched at a rate of 1.3 nm/cycle¹ and Ni₃Al at a rate of 1.0 nm/cycle, while NiAl and Al were not etched by this ALE process. Because films with high Al content did not etch, Ni clearly plays a role in the removal of Al. Possible volatile etch products of Al therefore include a dimeric complex containing both a Ni and Al atom, or trimethylaluminum from Al reacting with CH₃ formed by Ni-catalyzed hydrogenation of formic acid. Finally, the etch rate of blanket Ru films was determined to be 0.5 nm/cycle, demonstrating a 2:1 selectivity between Ni₃Al and Ru—a major advance toward integration of Ni₃Al in EUV masks.

¹T.G. Smith, A.M. Ali, J.F. de Marneffe, J.P. Chang, *JVST A* **42**, 022602 (2024).

6:00pm **AP+PS+TF-WeA-16 In-situ Comparative Analysis of Surface Reactions During Isotropic Atomic Layer Etching of TiC under Various Plasma Chemistries and Infrared Heating**, *Kazunori Shinoda*, Hitachi High-Tech Corporation, Japan; *Thi-Thuy-Nga Nguyen*, Nagoya University, Japan; *Dai Ishikawa, Kenetsu Yokogawa, Masaru Izawa*, Hitachi High-Tech Corporation, Japan; *Kenji Ishikawa, Masaru Hori*, Nagoya University, Japan

As integration density increases, field-effect transistors (FETs) have evolved from two-dimensional planar structures to three-dimensional FinFETs, with gate-all-around (GAA) structures now emerging at advanced technology nodes. To enable continued scaling, complementary FETs (CFETs) are being investigated as the next step beyond GAA. This structural progression has intensified interest in isotropic atomic layer etching (ALE), which offers atomic-level dimensional control critical for fabricating advanced three-dimensional semiconductor structures. In response to this demand, a thermal-cyclic ALE process has been developed, incorporating cyclic plasma exposure and infrared (IR) heating for surface modification and layer removal. A 300-mm dry chemical removal (DCR) tool was engineered to implement this process. Using this system, selective isotropic ALE has been demonstrated for materials including Si₃N₄, SiO₂, TiN, W, and Co. This study presents a comparative analysis of surface atomic-layer reactions during TiC ALE using various fluorocarbon-based plasma chemistries. In-situ X-ray photoelectron spectroscopy (XPS) was employed to characterize surface chemical bonding states, while in-situ spectroscopic ellipsometry was used to monitor etching performance during cyclic processing. The experimental setup comprised a radical irradiation chamber and an XPS analysis chamber, connected via a high-vacuum transfer system to preserve surface integrity. Exposure to radicals generated from fluorocarbon-based gas mixtures resulted in the formation of surface-modified layers containing Ti-F bonds. Exposure to radicals generated from nitrogen-containing fluorocarbon-based gases led to the appearance of N-H bonds, with a binding energy centered at 402 eV. This reaction pathway is analogous to that observed in TiN ALE, where the surface-modified layer was tentatively identified as containing ammonium salt-based species. These layers were effectively removed by IR heating. Repetitive cycles of plasma exposure and IR heating produced a linear increase in TiC etching depth with cycle count. The etch per cycle (EPC) varied with plasma chemistry, typically ranging from 0.9 to 1.4 nm/cycle under optimized conditions. Saturation behavior with respect to radical exposure time confirmed the self-limiting nature of the process. Throughout the ALE cycles, the etched TiC surfaces remained smooth.

Electronic Materials and Photonics

Room 207 A W - Session EM1+AP+CPS+MS+PS+SM+TF-WeA

Materials and Devices in Emerging Memories

Moderators: M. David Henry, Sandia National Labs, Philip Lee, University of Kentucky

2:15pm EM1+AP+CPS+MS+PS+SM+TF-WeA-1 Impact of Precursor Purge Time on the Performance of Ferroelectric Hf_{0.5}Zr_{0.5}O₂ Prepared by Plasma-Enhanced Atomic Layer Deposition, Yong Kyu Choi, Benjamin Aronson, Megan Lenox, Liron Shvilberg, University of Virginia, USA; Chuanzhen Zhou, North Carolina State University; Kristina Holsgrove, Queen's University Belfast, UK; Amit Kumar, Queen's University Belfast, UK; Andrea Watson, Stephen J. McDonnell, Jon F. Ihlefeld, University of Virginia, USA

Hafnium oxide (HfO₂) shows significant potential for non-volatile memory and energy harvesting applications. However, its monoclinic phase lacks polarization, making it unsuitable for ferroelectric applications. Introducing ZrO₂ into HfO₂ (HZO) helps stabilize a ferroelectric phase. Atomic layer deposition (ALD) is the most widely used film processing technique, offering excellent thickness control, conformability, and relatively low processing temperature. Previous research has explored the impact of various metal precursors, oxidizer precursors, and process temperatures on the ferroelectric properties of HZO. One common observation is that the metal precursor purge time has a large effect on the resulting film phase and performance. However, no clear mechanism has been identified to explain this effect. In this presentation, we will discuss how HZO thin film properties change when the metal precursor purge time varies during plasma-enhanced ALD. Reducing the metal precursor purge time from 90 s to 3 s induced a transition from ferroelectric to antiferroelectric properties with double polarization hysteresis loops, higher endurance and polarization stability, and slightly increased in relative permittivity. Infrared spectroscopy measurements (FTIR-ATR) confirmed that the antiferroelectric properties are due to the antipolar orthorhombic o-I phase, which is consistent with observations from HRTEM and DPC-STEM. The films deposited with shorter purge times showed carbon impurities as identified by ToF-SIMS analysis. This suggests that residual chemical ligands from incomplete precursor removal during the ALD process, in part, stabilizes the antipolar o-I phase. These results show that phase stability in fluorite oxides is influenced by impurities beyond intentional substituents and that stable antiferroelectric responses can be achieved without deliberately altering the material composition, such as adjusting the Hf:Zr ratio to control phase formation.

2:30pm EM1+AP+CPS+MS+PS+SM+TF-WeA-2 Effect of Atomic Layer Annealing Duration on Phase Stabilization of Hafnium Zirconium Oxide Thin Films, Nicolas Lam, University of Virginia; Gerald Bejger, John Barber, Virginia Tech; Megan Lenox, Liron Shvilberg, University of Virginia; Christina Rost, Virginia Tech; Jon Ihlefeld, University of Virginia

Significant research has gone into understanding the stabilizing mechanisms and properties of ferroelectric hafnia. This is largely due to its ability to display ferroelectricity in size scales below 10 nm, incorporation in already existing mass production infrastructure, and complementary metal oxide semiconductor compatibility. Today, hafnium zirconium oxide (HZO) is the most studied hafnia alloy due to its low processing temperature. However, the widespread implementation of HZO as a memory material is hindered by a variety of challenges, such as wake-up, imprint, and retention. A major issue is the inability to make phase pure ferroelectric HZO, a metastable non-centrosymmetric polar orthorhombic structure. Commonly cited impurity phases include the metastable tetragonal, antipolar orthorhombic, and equilibrium monoclinic phases. Previous work using the atomic layer annealing (ALA) technique has shown enhanced crystallinity and remanent polarization in pristine HZO films, circumventing significant formation of the antiferroelectric and tetragonal phases. In this work, thin films of HZO were grown using the ALA technique with various ALA treatment durations, ranging from 0 s up to 59 s. Following a deposition of a metal oxide layer using plasma-enhanced atomic layer deposition, the surface of the film was subjected to additional argon plasma. After synthesis and a post-metallization anneal to form the metastable phase, various structural and electrical measurement techniques were used to characterize the films. Grazing-incidence X-ray diffraction shows no formation of the equilibrium monoclinic phase; Fourier transform infrared spectroscopy shows increasing ferroelectric phase concentration with ALA time. Polarization hysteresis measurements show

an increasing hysteretic response with ALA time as compared to an antiferroelectric reference sample. Positive up negative down measurements quantified the relative amount of wake-up. The reference devices displayed a 200% increase in remanent polarization while the ALA samples displayed an 8% relative increase with the longest treatment time. The results suggest that ALA can modify the local environment of the deposited films, such that the phase fraction of the ferroelectric phase and the amount of wake-up can be tuned. This results in devices that exhibit minimal to no wake-up. This work furthers the understanding of the effect that ALA has on the resultant film's properties.

2:45pm EM1+AP+CPS+MS+PS+SM+TF-WeA-3 Understanding Time-Dependent Imprint in Hafnium Zirconium Oxide Based Ferroelectric Tunnel Junctions, Megan Lenox, University of Virginia, USA; Samantha Jaszewski, Sandia National Laboratories; Jon Ihlefeld, University of Virginia, USA; M. David Henry, Sandia National Laboratories, USA

While research into understanding the performance-materials property relationship of hafnium zirconium oxide (HZO) based devices has been accelerated in the past decade, their integration into microelectronic products is challenged by their endurance and imprint behavior. Imprint, or a shift in the coercive field following polarization with an initial applied field, lowers HZO remanent polarization (P_r) along the imprint direction, impacting the current transport mechanisms and reducing the overall performance stability when studied in ferroelectric non-volatile memory applications. In these devices, imprint has been hypothesized to result from charge carrier migration at the electrode interface, increasing the charge needed for polarization switching. However, the mechanisms responsible for imprint in ferroelectric tunnel junctions (FTJ) is not understood. To study FTJ imprint phenomena, 7 nm Hf_{0.7}Zr_{0.3}O₂ devices with NbN and Nb as the top and bottom electrode, respectively, were fabricated. Polarization-electric field measurements were performed every 2nd seconds, showing a + V_c shift with time.

Resistance measurements, using a pulsing scheme composed of a $\pm V_{max}$ write pulse followed by fifty 0.4 V read pulses at various pulse widths taken every 2nd seconds, showed a drift in the ratio of high and low resistance states, and an overall reduction in the binary state memory window with increasing time, characteristic of imprint. Further, these results highlight imprint impacts on multi-state polarization switching used in neuromorphic memory applications. To investigate imprint mechanisms, pulsed hysteresis measurements taken in 0.1 V intervals followed by a reset pulse at $\pm V_{max}$ showed an 18.1x change in the resistance ratio between the high and low resistance states. However, a similar pulsed hysteresis measurement without the reset pulse had a 11.4x resistance ratio. These results support the generation of time-dependent imprint-free HZO-based FTJs by utilizing selective pulsing schemes, promoting their use in next-generation microelectronics.

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc. for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. This work is supported by the Center for 3D Ferroelectric Microelectronic Manufacturing (3DFEM2), an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences Energy Frontier Research Centers program under Award Number DE-SC0021118.

3:00pm EM1+AP+CPS+MS+PS+SM+TF-WeA-4 Disentangling Gamma-Ray Radiation Effects and Time-Dependent Imprint on Ferroelectric Hafnium Zirconium Oxide-Based Devices, Samantha Jaszewski, Sandia National Laboratories; Megan Lenox, Jon Ihlefeld, University of Virginia; M. David Henry, Sandia National Laboratories

Ferroelectric hafnium oxide (HfO₂) enables technological developments in microelectronics, such as the scaling of ferroelectric random-access memory (FeRAM) and new devices like ferroelectric field-effect transistors (FeFETs) and ferroelectric tunnel junctions (FTJs) that were not previously possible with conventional ferroelectrics. This is due to the material's compatibility with silicon and its ability to exhibit a ferroelectric response in films as thin as 1 nm. Understanding the interaction between radiation and ferroelectric HfO₂-based devices is necessary before these devices can be utilized in radiation-hostile environments. In the literature, it has been reported that gamma-ray radiation can result in a shift of the coercive voltage of ferroelectric HfO₂-based devices, impacting the memory window and, thus, the reliability of these devices. However, ferroelectric HfO₂-based capacitors have also been shown to exhibit a time-dependent imprint effect in which the coercive voltage shifts over time as a result of the

depolarization field in the film, which drives charge redistribution in the ferroelectric layer. As such, it can be challenging to disentangle the effects of gamma-ray radiation and the time-dependent imprint shift when evaluating the performance of these devices.

In this work, ferroelectric hafnium zirconium oxide (HZO) capacitors and ferroelectric tunnel junctions (FTJs) are subjected to 1 and 5 Mrad doses of gamma-ray radiation under grounded and biased conditions. X-ray diffraction and Fourier-transform infrared spectroscopy measurements demonstrate that gamma-ray radiation does not result in phase transformations, further confirmed by capacitance-voltage measurements, which show that the relative permittivity of the HZO capacitors does not change after radiation. Polarization-electric field measurements show shifts in the coercive field after radiation. However, it will be shown that these coercive voltage shifts are due to time-dependent imprint in the material rather than the effects of gamma-ray radiation. This work demonstrates that the structural and electrical properties of ferroelectric HZO-based capacitors and FTJs are not affected by gamma-ray radiation up to doses of 5 Mrad. It also underscores the importance of careful measurement procedures and analysis when evaluating radiation effects in this material.

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Plasma Science and Technology Room 201 ABCD W - Session PS1-WeA

Plasmas for Emerging Device Technologies

Moderators: Michael Gordon, University of California at Santa Barbara, Kenji Ishikawa, Nagoya University, Scott Walton, Naval Research Laboratory

2:15pm **PS1-WeA-1 Main Etch Challenges in the GaN-based Devices**, Patricia Pimenta Barros, Simon Ruel, Univ. Grenoble Alpes, CEA, LETI, France; David Cascales, Univ. Grenoble Alpes, CEA, Leti and CNRS, LTM, France; Nicolas Posseme, Univ. Grenoble Alpes, CEA, LETI, France; Thoeille Philippe, Lam Research, France; Eugénie Martinez, Univ. Grenoble Alpes, CEA, LETI, France; Bassem Salem, Univ. Grenoble Alpes, CNRS, LTM, France; Maxime Pezeril, Khatia Benotmane, Univ. Grenoble Alpes, CEA, LETI, France; François Gaucher, Lam Research, France; Laura Vauche, Yveline Gobil, Univ. Grenoble Alpes, CEA, LETI, France

INVITED

Thanks to the inherent properties of Gallium Nitride, the semiconductor industry envisages the introduction of GaN in a wide range of applications. For instance, GaN-based high electron-mobility transistors (HEMTs) have been adopted in power devices thanks to their high breakdown electric field and electron mobility [1]. Also, GaN's direct wide-band gap (3.4eV) is exploited into LED, microLED and displays for better photon emission.

Among the manufacturing steps of GaN-based devices, the plasma etching steps are part of the most critical ones as they have to satisfy morphological requirements without damaging the GaN material. Indeed, when patterning the GaN-based HEMTs, the electrical performances are directly linked to the damage induced by plasma etching at the gate bottom [2]. Depending on the architectures, the GaN etching step has to comply with different morphological criteria: i) high pGaN etching selectivity over AlGaN in pGaN gate transistors, ii) vertical profiles with bottom rounded corners in recessed-gate transistors.

This talk will focus on the main etching challenges that occur during the gate patterning of GaN-based HEMTs, and will give an overview of our recent outcomes. First, a GaN etching mechanism with a resist and SiN hardmask will be proposed based on morphological studies and the chemical analysis of the remaining byproducts on GaN sidewalls analyzed. The best etch parameters leading to vertical GaN sidewalls and bottom rounded corners with an etched-depth of 1µm will be shared.

In the case of pGaN gate structure, Cl₂/N₂/O₂ and BCl₃/SF₆ based chemistries will be compared in terms of selectivity and profile. Secondly, the damage induced by plasma etching on the GaN surface was investigated. Thus, electrical characterizations have been conducted using either sheet resistance (R_{sheet}) or C-V measurements in order to simulate the pGaN and recessed-gate MOS transistors' behavior, respectively. The goal will be to compare the benefits and drawbacks of different Cl₂-based

etching processes, and to identify the main degradation mechanisms.

In conventional etching processes, it has been shown that passivating chemistries like SiCl₄-based processes could be an alternative solution for improving recessed gate-MOS transistors [3]. In addition, we demonstrated that Atomic Layer Etching (ALE) reduces the damage induced by conventional etching [4]. Finally, this paper will compare ALE and bias pulsed processes.

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2:45pm **PS1-WeA-3 Study of N-Polar GaN Etching by a CH₄/H₂/Ar Plasma for µLED Applications**, Sandra Kozuch, Simon Ruel, David Vaufray, Olivier Renault, CEA-Leti, France

The ability of Gallium Nitride (GaN) to form ternary alloys with Al or In for emission wavelength modulation makes it a material of choice for µLED (micro-Light Emitting Diodes) applications. The µLED studied structures are VTF (Vertical Thin Film) type implying the report of the GaN stack on a backplane resulting in a N-polar GaN exposed. The pixel fabrication involves a plasma etching step (mostly with a Cl₂-based chemistry), known to damage the material mostly at mesa sidewalls [1]. For instance, defects like lattice amorphization, nitrogen depletion, implantation or deposition of etching by-products can be responsible for non-radiative recombinations of electron-hole pairs. This phenomenon is heightened for smaller pixels and results in an efficiency loss for the devices with miniaturization.

To address these issues and improve device performances, there is a need to develop less damaging etch processes. A change of etching chemistry for CH₄/H₂ mix, and avoid using Cl₂, can be an interesting and still poorly investigated strategy: by-products formed by CH₄ and GaN are very volatile, preventing them from redepositing on the etched surface [2]. Moreover, H atoms can passivate donor states near the surface [3].

In this study, we propose to etch N-polar GaN with CH₄/H₂/Ar plasma in an ICP chamber, with the goal of understanding its etch mechanisms and impact on material degradation. Different etch parameters are studied as DC bias voltage, source power, pressure or gas ratio to found a maximum ER of 50 nm/min and 85° profile, as vertical sidewalls as for a chlorine-base etching.

Scanning Electron Microscopy (SEM) measurements enabled measuring N-polar GaN etching rate, carbon-containing by-products deposition rate and sidewalls verticality. The surface etched using the best conditions were first studied by X-Ray Photoelectron Spectroscopy to obtain N/Ga stoichiometry and study valence and core-level states to retrieve band bending and chemical bonding states. Secondly, cathodoluminescence was performed on the same samples to study the GaN Yellow Band emission (between 500 and 700 nm) linked to radiative defects emissions. Then, the results were compared with those of a Cl₂-based etch of reference. These characterizations aim to determine if the developed etching process is less invasive or not to the material.

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Wednesday Afternoon, September 24, 2025

Plasma Science and Technology

Room 201 ABCD W - Session PS2-WeA

Atmospheric Plasma

Moderators: Michael Gordon, University of California at Santa Barbara, Kenji Ishikawa, Nagoya University, Scott Walton, Naval Research Laboratory

3:00pm PS2-WeA-4 Investigating the Thermal Behavior of Atmospheric Pressure Plasma Jets on Different Surface Types, Vladimir Milosavljevic, School of Physics, Clinical & Optometric Sciences, Technological University Dublin, Ireland & Faculty of Physics, University of Belgrade, Serbia, Ireland; James Lalor, School of Physics, Clinical & Optometric Sciences, Technological University Dublin, Ireland

Atmospheric pressure nonthermal plasmas hold great promise for applications in environmental management, energy transformation, and material engineering. Although they operate at room temperature, nonthermal plasmas produce highly reactive species that can modify surfaces at the plasma/surface interface. This study examines the interaction of an Argon atmospheric pressure plasma jet (APPJ) with both insulating and conductive mesh surfaces. The dielectric barrier discharge APPJ functioned at 8 kV and 21 kHz.

Previous research has analyzed how an atmospheric pressure plasma jet behaves when directed perpendicularly onto both dielectric and conductive flat surfaces, revealing that the jet maintains a laminar flow, expanding radially from the impact point. The highest temperature occurs at the central impact zone, with a radial decrease outward due to jet expansion and heat dissipation along the surface.

In contrast, this study introduces a novel method by treating a mesh substrate with 0.8 mm x 0.8 mm openings, allowing partial gas plume penetration. This enables thermal mapping of the interaction between the APPJ and the substrate, offering insights into the jet plume's thermal cross-section. A series of experiments explored how different materials, such as metals and polymers, respond to the APPJ's thermal energy by analyzing temperature rise, heat distribution, and cooling rates. The distance between the APPJ nozzle and the mesh surface (standoff distance) was adjusted from 0 to 70 mm, with thermal profiles recorded to identify the optimal distance for preventing surface overheating. Additionally, treatment time was varied between 0 and 240 seconds at a fixed standoff distance to evaluate thermal effects over different exposure durations.

A FLIR i7 thermal camera with a 140 x 140-pixel resolution was employed to capture precise thermal images, enabling detailed measurement of temperature gradients across treated surfaces. Its high accuracy and sensitivity were crucial for assessing the APPJ's thermal impact on various materials, ensuring reliable data acquisition throughout the study.

This research investigates the thermal behavior of APPJ treatments on metallic and polymeric surfaces, emphasizing the effects of standoff distance and treatment duration. The results indicate that steel, with its high thermal conductivity, heats and cools rapidly, whereas polypropylene retains heat longer due to slower heating. Findings also demonstrate that reduced standoff distances increase energy transfer, with material properties playing a crucial role in temperature distribution.

3:15pm PS2-WeA-5 Controlling Nitrogen Product Distributions in Plasma Electrolytic Reactors for Microbial Growth, Brandon Kamiyama, Diep Nguyen, Mohammadali Eslamisaray, Emily Gillmore, Angela Tomita, Ting Lu, R. Mohan Sankaran, University of Illinois at Urbana Champaign

Fixed forms of nitrogen are essential for the growth of plants that enable global food production, and for the growth of microorganisms which power critical processes beyond agriculture such as biomanufacturing and chemical production. Currently, nitrogen fixation is predominantly carried out by industrial processes (e.g., Haber-Bosch, Ostwald processes) that have large physical and environmental footprints. The development of alternative methods that are sustainable and deployable at a small scale for point-of-use production has emerged as one of our critical technological challenges. Among the different approaches being explored, plasmas in contact with liquids have shown great promise, capable of reacting nitrogen in air with water as a source of hydrogen at atmospheric pressure and near room temperature. However, a key challenge is that these processes generate many nitrogen products, including ammonium, nitrate, and nitrite ions, in addition to other products such as hydrogen peroxide.

In this work, we studied a direct-current plasma-based electrolytic reactor and correlated process conditions such as gas feed, pH, and electrode polarity with product yields and selectivity. In particular, molecular oxygen and pH were found to be key for controlling the selectivity between the

reductive and oxidative species. These results provided insight into possible reaction mechanisms and enabled us to selectively synthesize nitrogen products as substrates for microbial growth and biosynthesis.

3:30pm PS2-WeA-6 Particle-in-Cell Modeling of the Reverse Anode Sheath in Parallel-Plate Sub-Atmospheric Pressure Hydrogen Plasmas, Brian Jensen, Princeton University Plasma Physics Lab; David Graves, Princeton University

Parallel-plate electrode plasmas are widely used as large-area semiconductor processing tools [1-4]. In these systems, non-equilibrium electrons with non-Maxwellian electron energy distribution functions (EEDFs) drive gas-phase reactions and enable surface modification [5]. Particle-in-cell (PIC) simulations are well-suited to capture such effects, but at pressures above a few torr, 2D and 3D simulations with appropriate reactor chemistry become increasingly expensive due to disparities between plasma and chemical length and time scales. To investigate these systems, this work presents a coupled 1D PIC-fluid model for hydrogen plasma reactors and studies the effectiveness of DC parallel plate systems as tools for large-area processing, particularly for diamond substrates. We characterize the reverse anode sheath for systems with gas pressures exceeding 10 Torr and describe the range of unique processing conditions available at the anode surface under these conditions.

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Plasma Science and Technology

Room 201 ABCD W - Session PS3-WeA

ICP Modelling

Moderators: Thorsten Lill, Lam Research Corporation, Shahid Rauf, Applied Materials, USA

4:15pm PS3-WeA-9 Quantum Chemistry and Integrated Modeling for Understanding the Mechanisms of Selective and Cryogenic Atomic-Scale Etching, Yuri Barsukov, Mingmei Wang, Qing Xu, Thorsten Lill, Lam Research Corporation

INVITED

Plasma etching for high aspect ratio vertical trenching in 3D-structured silicon-based devices is one of the most challenging steps in advanced semiconductor manufacturing. This process requires precise control of both ion and neutral fluxes to facilitate etching at the trench bottom while ensuring sidewall passivation to prevent lateral etching and feature distortion. As the range of chemical reactants used in industry continues to expand, a deeper understanding of plasma-surface interactions and surface reaction mechanisms becomes increasingly critical. Over the past decade, quantum chemistry has played a growing role in elucidating these mechanisms, providing valuable insights for optimizing plasma etching processes.

Quantum chemistry is widely used to investigate reaction mechanisms at the atomic level. Within the framework of transition state theory, the reactivity of various fluorine-based reactants with semiconductor materials has been calculated, revealing how etching with these reactants can be catalyzed, enhanced, and accelerated through vibrational excitation. This ab-initio approach enables the calculation of rate constants for key surface reactions and allows for the integration of surface reactions kinetics with plasma chemistry models. These kinetic models predict the dependence of etching rates and selectivity on plasma parameters. For example, the reactivity of fluorine (F) atoms and hydrogen fluoride (HF) molecules – two of the most commonly used reactants in the semiconductor industry – has been studied on silicon-based materials such as Si, SiN, and SiO₂.

Another crucial challenge in plasma-assisted etching is the efficient delivery of ions to the trench bottom. Accelerated ions lose kinetic energy through the collisions with sidewalls, leading to feature damage without effectively contributing to bottom etching. Despite their high initial energies in the keV range, the normal component of ion energy at the grazing incident is only in tens of eV. As a result, relatively weak chemical interactions between

sidewall materials and incident ions play a crucial role in determining etching efficiency and feature integrity. Using ab-initio molecular dynamics, it has been demonstrated that ammonia fluoride ionic salts – the most common etching by-products that coat the sidewalls – provide more effective protection against damage and help prevent ion energy loss at lower temperatures. This discovery sheds light on the mechanisms of cryogenic plasma-assisted etching and highlights the importance of by-product formation in sustaining etching process.

4:45pm PS3-WeA-11 Simulation of an Inductively Coupled Plasma with a Two-Dimensional Darwin Particle-in-Cell Code, *Dmytro Sydorenko*, University of Alberta, Edmonton, AB, Canada; *Igor Kaganovich*, *Alexander Khrabrov*, Princeton Plasma Physics Laboratory

Electromagnetic simulation with an explicit algorithm has a severe limitation on the time step due to the large speed of light propagation resulting in the high numerical cost. Fully implicit electromagnetic algorithms do not have this limitation but are more complex to implement. Another option is the Darwin method omitting the electromagnetic wave propagation [1]. The Darwin method separates the electric field into solenoidal (electromagnetic) and irrotational (electrostatic) parts.

In this work, we propose a new Darwin scheme for simulation of low-frequency electromagnetic processes in laboratory plasmas. A two-dimensional particle-in-cell code in Cartesian geometry has been developed based on the direct implicit Darwin electromagnetic algorithm described in Ref. 1. The new code has several significant modifications compared to the original algorithm. First, the SDF is replaced by a new method based on the equation for the vorticity of the solenoidal electric field. Unlike the SDF, the linear system of equations in the vorticity method is reliably solved using a standard iterative solver. Second, the electromagnetic fields are defined on staggered grids convenient for electromagnetic simulation. Third, the contribution of collisional scattering is included in calculation of the solenoidal electric fields. Fourth, the code includes several solvers for the self-consistent magnetic field with different boundary conditions. Once one of these methods is selected for a particular simulation, the choice can be verified by checking the energy conservation.

A two-dimensional particle-in-cell code has been developed using the modified direct implicit Darwin electromagnetic algorithm described in Ref. 2. The code is a valuable tool for simulation of various electromagnetic effects, for example the inductively coupled plasmas and the electromagnetic plasma waves. The code can be used to design future plasma thrusters.

References:

[1] M. R. Gibbons and D. W. Hewett, "The Darwin Direct Implicit Particle-in-Cell (DADIPIC) Method for Simulation of Low Frequency Plasma Phenomena," *J. Comput. Phys.* 120, 231–247 (1995).

[2] Dmytro Sydorenko, Igor D. Kaganovich, Alexander V. Khrabrov, Stephane A. Ethier, Jin Chen, Salomon Janhunnen, "Improved algorithm for a two-dimensional Darwin particle-in-cell code", arXiv:2409.19559, submitted to *Phys. Plasmas* (2024).

5:00pm PS3-WeA-12 Exploring the Impact of Mask Geometries on High Aspect Ratio Silicon Etching Using Cl_2/O_2 Plasmas, *Shahid Rauf*, *Xingyi Shi*, *Han Luo*, *Jason Kenney*, *Geuntak Lee*, *Sonam Sherpa*, *Takumi Yanagawa*, Applied Materials

As computing technology advances, the demand for more intricate geometries in etching processes has surged, necessitating a deeper understanding of the underlying physics. While previous published computational studies predominantly focus on via and trench geometries, the challenges posed by alternative mask geometries remain largely unexplored. This study employs Monte Carlo-based feature scale simulations to investigate high aspect ratio silicon etching using Cl_2/O_2 plasma. Initially, we present the general behavior of etching features with a rectangular geometry to establish a baseline. Subsequently, we explore the influence of chemical composition and bias voltage pulsing on the etching profile, highlighting how these parameters can be optimized for improved precision and control. The study culminates in an analysis of the impact of mask geometry by comparing etching profiles produced with circular, square, and rectangular mask shapes. Our findings reveal significant variations in etching outcomes based on mask geometry, underscoring the need for tailored approaches in feature scale simulations. This research not only broadens the understanding of etching dynamics but also paves the way for more sophisticated design strategies in semiconductor manufacturing, addressing the evolving demands of modern computing technologies.

5:15pm PS3-WeA-13 Modeling of Remote Inductively Coupled Plasmas and Comparison to Experiments, *Mackenzie Meyer*, *David Boris*, *Michael Johnson*, *Jeffrey Woodward*, *Virginia Wheeler*, US Naval Research Laboratory; *Mark Kushner*, University of Michigan; *Scott Walton*, US Naval Research Laboratory

Plasma-enhanced atomic layer deposition (PEALD) utilizes plasma as a source of reactive species. Using plasma enables processing at low temperature and with materials that cannot be processed using thermal atomic layer deposition. Remote inductively coupled plasmas (ICPs) are utilized in PEALD as they limit damage to the substrate. Since the plasma is spatially removed from the substrate by 10s of cm, energetic ions are limited while radicals remain plentiful at the substrate location. However, questions remain about the physics of remote ICPs downstream of the plasma source. To help unravel the physics occurring in these devices, we model a remote ICP system using the 2D Hybrid Plasma Equipment Model (HPEM). The remote ICP system is based on the Veeco Fiji G2 source. We focus on pure Ar plasmas over a range of pressures and powers. Power is coupled both inductively and capacitively to the plasma. Based on the location of the powered end of the coil, the capacitively coupled power is deposited near the exit of the ICP and into the spatial afterglow. The results of the model are benchmarked against Langmuir probe measurements at these conditions. The effect of N_2 addition to the Ar plasma is also examined and benchmarked against measurements. These results are discussed in the context of PEALD.

This work is partially supported by the Naval Research Laboratory base program.

5:30pm PS3-WeA-14 Modeling of E-H Transition in Inductively Coupled Plasmas, *Ashish Sharma*, *Rochan Upadhyay*, *Sudharshanaraj Thirupathiraj*, *Dmitry Levko*, *Anand Karpatne*, *Radhika Mani*, Lam Research Corporation

E to H transition is a phenomenon observed in plasma discharges and has been known to have a significant impact on the plasma etching characteristics. In the present study, we investigate the phenomenon of E-H transition for inductively coupled plasmas. These simulations have been conducted for a 2D GEC RF Reference cell in Cl_2 gas using VizGlow®. We study the transition of the plasma discharge from E-mode to H-mode and investigate the underlying physics governing the transition. We quantify the percentage of the input power absorbed in E mode and H mode and study the influence of TCP power, coil frequency and gas pressure on the power breakdown and E-H transition characteristics. Lastly, we analyze the plasma properties in E and H mode, mainly focusing on the differences in plasma densities, electron temperature and ion fluxes in these respective modes.

5:45pm PS3-WeA-15 Fully Kinetic Modeling of ICP Chambers Used for Plasma Processing, *Daniel Main*, *Thomas Jenkins*, *Scott Kruger*, *John Cary*, Tech-X Corporation

Low-temperature kinetic plasma simulations using particle-in-cell (PIC) and Monte Carlo methods (DSMC/MCC) for the chemistry can provide many advantages over fluid simulations, including detailed information about the Ion Energy Distribution Function (IEDF) and Ion Angular Distribution Function (IADF) that are critical for plasma processing. In addition, a fully kinetic approach does not make common assumptions made in fluid models, such as local conductivity or Maxwellian distributions of the plasma species. In this talk we present kinetic modeling results of inductively coupled plasmas in a 2D cylindrically symmetric geometry. We demonstrate how implicit methods can make these challenging simulations feasible by reducing computing times by factors of 20-200. We also demonstrate a method of providing constant power to the plasma, which further decreases the runtime needed to achieve steady-state discharges. We then apply DC and/or RF bias voltage below the wafer, introducing capacitive coupling self-consistently into the model to enable better etch control, and explore how steady-state ion fluxes and IEDF/IADFs at the wafer surface vary as a function of RF bias frequency, amplitude, and waveform shape. We show, for example, that a low-frequency CCP bias couples more efficiently with the ions leading to an increase in the RF-averaged ion energy. We also demonstrate that improved IEDF uniformity can be achieved through careful choice of the shape of the bias waveform.

6:00pm PS3-WeA-16 Comparative Analysis of Methods to Obtain EEDF in Plasma Simulations for Semiconductor Processing, *Chenhui Qu*, *Matt Talley*, *Saravanapriyan Sriraman*, Lam Research Corp.

Accurate determination of the Electron Energy Distribution Function (EEDF) is crucial for modeling plasma behavior and predicting gas-phase chemistry in semiconductor processing. Understanding the EEDF allows for precise control over plasma characteristics, essential for optimizing semiconductor

Wednesday Afternoon, September 24, 2025

manufacturing hardware and process technology. There are three primary methods for obtaining EEDF in plasma simulations: (a) kinetic particle approach using Monte Carlo, (b) the inline Boltzmann solver, and (c) the Maxwellian approximation, each with distinct advantages and limitations based on plasma conditions.

The kinetic approach offers the highest accuracy by treating electrons as particles and resolving electron trajectories under electromagnetic fields, capturing non-Maxwellian distributions and spatial variations. It provides detailed insights but is computationally intensive and may be prone to statistical noise.

At the farthest extreme, the Maxwellian approximation, while computationally efficient, assumes the electrons to be in thermal equilibrium and often fails to represent complex plasma behaviors accurately. While it reduces computational requirements, its applicability is limited and may not provide the necessary accuracy for advanced semiconductor processing at low pressures.

In the middle of the spectrum, the inline Boltzmann solver approach trades off accuracy to efficiency by solving the Boltzmann equation, in typically two-term or “few-term” expansion. The method offers a compromise between the kinetic approach and Maxwellian approximation, but can struggle under extreme non-equilibrium scenarios.

In this presentation, case studies of Inductively Coupled Plasma (ICP) and Capacitively Coupled Plasma (CCP) that demonstrate the impact of different approaches in obtaining EEDFs on plasma distributions, and ultimately the on-wafer results will be covered. Comparisons to experiments that validate the kinetic approach’s superiority will be discussed highlighting the stringent standards required in semiconductor industry plasma simulations.

Atomic Scale Processing Mini-Symposium

Room 206 A W - Session AP+AS+EL+EM+PS+TF-ThM

Advancing Atomic Scale Processing through Modeling and Simulation

Moderator: Sagar Udyavara, Lam Research Corp

8:00am **AP+AS+EL+EM+PS+TF-ThM-1 Multiscale Simulations for Atomic Scale Processing**, *Michael Nolan*, Tyndall Institute, Ireland **INVITED**

In modern semiconductor device fabrication, the dimensions involved require atomic level control over materials deposition and etch. Atomic Level Processing, exemplified by Atomic Layer Deposition (ALD) and thermal atomic layer etch (tALE), is therefore critical deposition and etch of relevant materials. Further scaling and use of complex three-dimensional structures means that Thermal ALE will take centre stage in etching. The key chemistry takes place at surfaces which drives the self-limiting characteristics and other advantages of these atomic level processing approaches. In this presentation I will discuss how atomistic simulations based on first principles Density Functional Theory, ab initio Molecular Dynamics and kinetic Monte Carlo methods can be used to predict the chemistry of atomic level deposition and etch processes. I will first discuss the key chemistries involved in atomic level processing chemistries and the challenges that we have identified in this exciting area. The first scientific topic is the simulation of plasma enhanced deposition (PE-ALD) of metals, using the example of cobalt for next generation interconnects. This is the first example of an atomistic level study of the full PE-ALD cycle for Co metal and show that the process requires use of ammonia or mixed H₂/N₂ plasma. Calculated energy barriers for key steps give guidance regarding the temperatures required for the process. We show how substrate pre-treatment can reduce nucleation delay and therefore allow selectivity in deposition of the target film. Finally we show how kinetic Monte Carlo can be used to predict the structure of deposited metal films on different nitride substrates using data from DFT level simulations. The second example is molecular layer deposition of hybrid materials, using alucone as the prototypical example. Comparison of aliphatic with functionalized aromatic molecules allows differences in film properties to be understood. A further application of this involves selective, templated deposition of target films using block co-polymer infiltration where differences in reactivity of a precursor in two polymers promotes selective deposition of the target films. Finally, I present our work on self-limiting thermal atomic layer etching (ALE), highlighting how simulations can (1) predict the window of self-limiting etch (2) unravel the difference between amorphous and crystalline substrates and (3) probe the impact of surface orientation on tALE chemistry, all of which are important for future, selective thermal ALE processing on complex 3D substrates.

8:30am **AP+AS+EL+EM+PS+TF-ThM-3 The Si-Cl₂-Ar⁺ Atomic Layer Etching Window: Fundamental Insights from Molecular Dynamics Simulations and a Reduced Order Model**, *Joseph Vella*¹, TEL Technology Center, America, LLC, USA; *David Graves*, Department of Chemical and Biological Engineering Princeton University

Plasma assisted atomic-layer etching (ALE) processes are frequently characterized by the ALE window. This is a range of ion energies where the amount of substrate etched remains constant as a function of the ion energy. Silicon (Si) etch by alternating exposure to chlorine gas (Cl₂) and argon ions (Ar⁺) is frequently used as a demonstrative example to illustrate concepts of ALE, including the ALE window.[1] Despite this, when examining the literature, properties of the ALE window for this system remain obscure. For example, Kim et al.[2] studied Si-Cl₂-Ar⁺ ALE and report that the ALE window should be below 40 eV. On the other hand, Park et al.[3] report the ALE window as being from 70 to 90 eV. Still others report an Ar⁺ ion energy of 50 eV as being within the ALE window.[4] In this talk, we aim to resolve these contradictory reports by studying the Si-Cl₂-Ar⁺ ALE with classical molecular dynamics (MD) simulations and a reduced order model (ROM).[5] The MD results show that the range of Ar⁺ ion energies where the amount of Si etched per cycle (EPC) remains relatively constant is from 15eV to 20 eV, which is very narrow. The EPC in this region is also less than one atomic layer, because atomic Cl sputtering is significant. The results also show that a large ion fluence (roughly 4.2 10¹⁶ ions/cm² for 15 eV ions) is required to remove all Cl from the near surface region, which is a key insight when developing processes that achieve “true ALE”. Using the

ROM, parameters can be varied to observe their effect on properties of the ALE window. For example, by increasing the threshold sputtering energy of Si, the width of ALE window can be increased. While this study focuses on the relatively simple Si-Cl₂-Ar⁺ system, it is clear learnings from this study can be extended to other systems.

References

- [1] T. Lill, “Atomic Layer Processing: Semiconductor Dry Etching Technology” (Wiley-VCH, Weinheim, 2021).
- [2] B. Kim, S. Chung, and S. M. Cho, “Layer-by-layer Etching of Cl-adsorbed Silicon Surfaces by Low Energy Ar⁺ Ion Irradiation”, Appl. Surf. Sci. 2002, 187, 124-129.
- [3] S. Park, K. Min, B. Yoon, D. Lee, and G. Yeom, “Precise Depth Control of Silicon Etching using Chlorine Atomic Layer Etching” Jpn. J. Appl. Phys. 2005, 44, 389-393.
- [4] K. J. Kanarik, T. Lill, E. A. Hudson, S. Sriraman, S. Tan, J. Marks, V. Vahedi, and R. A. Gottscho, “Overview of Atomic Layer Etching in the Semiconductor Industry”, J. Vac. Sci. Technol. A, 2015, 33, 020802.
- [5] J. R. Vella, Q. Hao, M. A. I. Elgarhy, V. M. Donnelly, and D. B. Graves, “A Transient Site Balance Model for Atomic Layer Etching”, Plasma Sources Sci. Technol., 2024, 33, 075009.

8:45am **AP+AS+EL+EM+PS+TF-ThM-4 Influence of Fluorination and Oxygenation Sources on the Thermal Atomic Layer Etching of MoS₂**, *Spencer Smith*, *Jacob A. Tenorio*, *Icelene Leong*, *John D. Hues*, *Steven M. Hues*, *Elton Graugnard*, Boise State University

Atomic layer etching (ALE) has emerged as a pivotal technique in the precise fabrication of two-dimensional (2D) materials, particularly molybdenum disulfide (MoS₂), which holds promise in the semiconductor industry due to its high mobility in monolayer form. The ability to precisely etch amorphous and crystalline MoS₂ films provides a pathway for controlling thickness, which is critical to achieving desired electrical and optical properties. Previous studies used MoF₆ and H₂O in thermal ALE of MoS₂. Here, we report studies of alternate sources of fluorination and oxygenation and evaluate their impact on thermal ALE of MoS₂. Oxygen sources include water and ozone, and fluorine sources include HF/Pyridine and MoF₆. Etch rates, uniformity, and surface chemistry post ALE were characterized using spectroscopic ellipsometry, atomic force microscopy, and X-ray photoelectron spectroscopy. Results indicated at ALE of amorphous MoS₂ with HF with either H₂O or O₃ showed no signs of etching at 200 °C or 250 °C. Whereas the combination of MoF₆ + O₃ at 250 °C on amorphous MoS₂ films exhibited an etch rate of 1.6 Å/cycle and a mass loss of 44 ng/cm². Further MoF₆ + O₃ etching at 200 °C showed a mass loss of 19 ng/cm², similar to prior reports using MoF₆ + H₂O at 200 °C. Surface morphology showed little change from etching, but surface oxygen concentration increased. This research further expands the capabilities for atomic layer processing of 2D materials.

9:00am **AP+AS+EL+EM+PS+TF-ThM-5 Insights Into Atomic Layer Etching of Diamond Surfaces**, *Jack Draney*, *Athanassios Panagiotopoulos*, *David Graves*, Princeton University

Thanks to its nitrogen vacancy color centers, diamond is a candidate for many quantum applications from quantum sensing to quantum computing. Pristine surfaces engineered for each application are required for good device performance. We investigated atomic-scale plasma processing as a method for reaching these pristine diamond surfaces. Our investigation takes the form of combined experiments and molecular dynamics simulations, allowing atomic-scale insights into the effects of argon / oxygen atomic layer etching on diamond surfaces.

9:15am **AP+AS+EL+EM+PS+TF-ThM-6 Benchmarking Large Language Models for Atomic Layer Deposition**, *Angel Yanguas-Gil*, *Matthew T. Dearing*, *Jeffrey W. Elam*, *Jessica C. Jones*, *Sungjoon Kim*, *Adnan Mohammad*, *Chi Thang Nguyen*, *Bratin Sengupta*, Argonne National Laboratory

In this work we introduce an open-ended question benchmark, ALDBench, to evaluate the performance of large language models (LLMs) in the field of atomic layer deposition. Our benchmark comprises questions with a level of difficulty ranging from graduate level to domain expert current with the state of the art in the field. Human experts reviewed the questions along the criteria of difficulty and specificity, and the model responses along four different criteria: overall quality, specificity, relevance, and accuracy. We ran this benchmark on an instance of OpenAI's GPT-4o using an API interface. This allows us to fine tune hyperparameters used by the LLM for text

¹ JVST Highlighted Talk

generation in a way that is not possible using conventional chat-based interfaces.

The responses from the model received a composite quality score of 3.7 on a 1 to 5 scale, consistent with a passing grade. However, 36% of the questions received at least one below average score. An in-depth analysis of the responses identified at least five instances of suspected hallucination. We also observed statistically significant correlations between the following question and response evaluation criteria: difficulty of the question and quality of the response, difficulty of the question and relevance of the response, and specificity of the question and the accuracy of the response. Finally, we will address other issues such as reproducibility, impact of hyperparameters on the quality of the response, and possible ways in which the performance of the LLMs can be further improved.

[1] A. Yanguas-Gil et al, *J. Vac. Sci. Technol. A* 43, 032406 (2025)

9:30am **AP+AS+EL+EM+PS+TF-ThM-7 Developing a “Digital Twin” for Area-Selective Deposition on 3D Nanopatterns**, *Nicholas Carroll, Gregory Parsons*, North Carolina State University

Area-selective deposition (ASD)—a bottom-up patterning technique that enables precise material deposition on specific regions while preventing deposition elsewhere—has garnered significant attention as an augmentation to lithographic patterning of nanoscale features during semiconductor manufacturing. Some potential applications, such as contact-over-active-gate, will require multiple ASD materials to be deposited in sequence, heightening the challenge of effective process design. Given the vast time and resources required for experimental assessments of process integration, demand is rapidly growing for a “digital twin” (i.e. a software representation of a physical system) of device fabrication sequences. A comprehensive ASD digital twin will require advances in analyzing atomic layer deposition (ALD) reactor design and mechanistic insights into interactions between inhibitor molecules, ALD reactants, and substrate surfaces over time as reactions proceed.

We have recently developed a stochastic lattice model describing metal oxide ASD on planar substrates, including means to visualize the film shape and extent of lateral overgrowth during ASD.^[1] Parameters in the model can be adjusted based on steric hindrance during each half-cycle, differences in interfacial energies between the non-growth surface and the growing film, and the preferred molecular bonding orientations. These factors elucidate subtleties in shape evolution during ASD, but results to date have been limited to vertical and lateral growth on 2D surfaces. A functional ASD digital twin must describe ASD on arbitrary 3D nanopatterns and on sub-lithographic feature sizes, including effects of selectivity loss where the selectivity decreases as film thickness increases.

We will present recent efforts in our group to extend the functionality of the stochastic lattice model to describe ASD on 3D substrates, including surfaces with pattern dimensions less than 10 nm. On very small features, for example, the model shows that lateral growth during ASD results in a wide distribution of feature separation distances, even when the growth per cycle is uniform across a growing film surface. We will also discuss intricacies that need to be considered to integrate multiple ASD steps into processes involving more complex “multi-color” substrates where several substrate materials exposed to reactants simultaneously. We believe that such insight will be critical for the realization of a functional digital twin model of atomic-scale processing needed for future semiconductor devices and other advanced manufacturing processes.

(1) Carroll, N. M.; Parsons, G. N. *J. Vac. Sci. Technol. A* 42 (6), 062411 (2024).

9:45am **AP+AS+EL+EM+PS+TF-ThM-8 Activation of C-X Bonds on Transition Metal Surfaces: Insight from DFT Studies**, *Matias Picuntureo*, Universidad Tecnica Federico Santa Maria, Chile; *Ilker Tezsevin, Marc Merckx*, Eindhoven University of Technology, The Netherlands; *Scott Semproni, Jiun-Ruey Chen*, Intel Corporation; *Adriaan Mackus*, Eindhoven University of Technology, The Netherlands; *Tania Sandoval*, Universidad Tecnica Federico Santa Maria, Chile

Area-selective atomic layer deposition (AS-ALD) represents an advanced bottom-up nanofabrication technique enabling selective material growth on targeted areas of patterned substrates. In advanced semiconductor manufacturing, such as next-generation processes at the back end of line (BEOL), small molecule inhibitors (SMIs) can enable AS-ALD through the selective formation of inhibitor layers on metal surfaces that block deposition.

A recent study by Merckx et al. reported hydrogenolysis and potentially dehydrogenation of aniline on Ru surfaces during AS-ALD, leading to the formation of a carbonaceous layer with enhanced inhibition performance.

This highlights the importance of understanding the driving forces behind the surface chemistry of SMIs.

To explore whether similar surface-mediated reactions can occur for other inhibitor–metal combinations, we employ density functional theory (DFT) to investigate the adsorption and dissociation mechanisms of benzene-derived SMIs on Ru(0001), Mo(110), and W(110) surfaces.

To enable a systematic comparison across different molecules and surfaces, our study focuses on radical-mediated dissociation pathways involving the cleavage of functional groups from the aromatic ring. This approach allows us to isolate the effect of the functional group and its interaction with the metal surface in determining the reaction thermodynamics between the molecular and dissociated adsorbed states.

We find that charge transfer to the adsorbed inhibitor modulates its dissociation energy landscape. The resulting radical intermediates are substantially stabilized through coordination with the metal surface. We further explore their subsequent hydrogenation, which transforms these surface-bound radicals into more stable, saturated species. Lastly, we show that the fate of reaction by-products—whether they remain adsorbed or desorb into the gas phase—can significantly impact the overall reaction thermodynamics and shift the equilibrium toward or away from product formation.

The investigation of the reaction pathways explored in this study contributes to the fundamental understanding of molecule–surface interactions during AS-ALD and offers insight that may support future strategies for the rational design of small molecule inhibitors.

References:

[1] Merckx et al., *J. Chem. Phys.* 160, 2024.

11:00am **AP+AS+EL+EM+PS+TF-ThM-13 Descriptor-driven analysis of inhibitors for AS-ALD processes**, *Joost F. W. Maas, Marc J. M. Merckx*, Eindhoven University of Technology, Netherlands; *Matias Picuntureo, Lucas Lodeiro*, Universidad Tecnica Federico Santa Maria, Chile; *Adriaan J. M. Mackus*, Eindhoven University of Technology, Netherlands; *Tania E. Sandoval*^{1,2}, Universidad Tecnica Federico Santa Maria, Chile

Area selective atomic layer deposition (AS-ALD) is a bottom-up technique that can address some of the challenges that limit the nanofabrication of complex structures, which require patterning and alignment at the atomic scale. Currently, one of the most robust strategies to carry out AS-ALD is with the use of small molecule inhibitors (SMIs), that selectively adsorb and inhibit the non-growth surface (NGS) and prevent precursor adsorption. These SMIs range from a variety of functionalities and structures depending on the target NGS, and their selection is based on specific criteria, such as reactivity, volatility, and safety.^{1,2}

Currently, the library of tested inhibitor molecules is very limited, therefore finding the best candidate for a given surface is challenging. Using computational tools can significantly accelerate the expansion of this library through high-throughput screening and recent advances in machine learning. In the case of the use of descriptors,³ the goal is to correlate the performance of the SMIs e.g., measured in terms of their stability, as adsorption energy, with the dependence on materials or molecular properties. The derived correlations can serve to establish general guidelines for SMI selection, expanding the analysis to other molecules not included in the initial study. This approach has proven to be very successful in reducing computational costs in other fields, such as heterogeneous catalysis and drug discovery.

In this presentation, we provide an overview of the dependency between a list of descriptors and the adsorption energies of SMIs candidates on a variety of relevant NGS, such as oxides, nitrides, and metals. We explore descriptors based on the molecular properties, such as electronegativity, electrophilicity, and orbital energy, as well as descriptors based on the electronic structure of the material, such as d-band center. Results indicate a with strong correlation with the adsorption energy (E_{ads}) and electronegativity of the core-atom on the adsorption of oxides and nitrides, as well as the d-band center on the adsorption on metal surfaces. Moreover, our data highlights the differences in reactivity across surfaces and the challenges in surface passivation across surfaces with similar surface sites. Overall, this study provides important insights into the use of descriptor-driven analysis in the selection of the right SMI candidates for the advancement of ASD processes.

[1] A. Mameli and A. Teplyakov *Acc. Chem. Res.* 2023, 56, 2084–2095.

¹ TFD Paul Holloway Award Winner

² JVST Highlighted Talk

[2] P. Yu, et al. *Appl. Surf. Sci.* 2024, 665, 160141.

[3] C. Chen, et al. *J. Phys. Chem. C* 2025, 129, 13, 6245–6253.

11:15am **AP+AS+EL+EM+PS+TF-ThM-14 Understanding Plasma-Induced Bonding and Composition Changes in SiCN ALD via kMC–DFT Modeling**, **Ting-Ya Wang**, University of Texas at Austin; **Hu Li**, **Peter Ventzek**, Tokyo Electron America; **Gyeong Hwang**, University of Texas at Austin; **Jianping Zhao**, Tokyo Electron America

Plasma-enhanced atomic layer deposition (PEALD) enables low-temperature processing of silicon carbonitride (SiCN), a critical low-k material for advanced interconnects. However, energetic plasma species—including both ions and radicals—can significantly influence surface reactions, film composition, and structural evolution, ultimately affecting material properties such as dielectric constant and mechanical strength. A comprehensive understanding of these species-specific effects is essential for process optimization.

Integrating kinetic Monte Carlo (kMC) with density functional theory (DFT) offers a powerful approach for simulating ALD. However, a key challenge in kMC lies in the need for a predefined list of permissible events. Traditionally, researchers manually compile a set of reactions deemed most significant. Yet, the vast number of possible events on a surface, combined with the importance of rare events in ALD, raises concerns about the authenticity and completeness of outcomes derived from manually curated reaction lists.

To address this, we developed an atomistic, off-lattice, three-dimensional simulator that integrates kMC with DFT. We employed a strategic approach to construct a comprehensive event list, capturing a broad spectrum of potential surface reactions. This year, we expand our study in four key directions: (1) comparison of ion- and radical-driven reaction pathways to delineate their distinct roles in modifying surface chemistry; (2) evaluation of different plasma chemistries (e.g., N_2 vs. NH_3) to understand how reactive species impact film stoichiometry and termination; (3) simulation of multi-cycle growth to track the evolution of defects and compositional shifts; and (4) simulation predictions against experimental data such as XPS and IR spectra.

Our findings reveal a synergistic interplay between ions and radicals in shaping the formation of Si–N, Si–C, and C–N bonding networks. The simulation platform enables insights into plasma–surface interactions, offering a predictive framework for optimizing SiCN PEALD processes.

11:30am **AP+AS+EL+EM+PS+TF-ThM-15 Understanding SiCN Film Oxidation Mechanism Through Density Functional Theory**, **Tsung-Hsuan Yang**, **Hu Li**, **Jianping Zhao**, **Peter Ventzek**, Tokyo Electron America

Low dielectric constant (low-k) spacers are essential components in advanced microelectronic devices for mitigating parasitic capacitance and crosstalk, leading to enhanced device performance. Among low-k materials, silicon carbon nitride (SiCN) is widely used for its tunability in dielectric constant, leakage current and chemical robustness. However, the long-term stability of SiCN films is often compromised by atmospheric moisture, leading to the formation of silicon oxide. To address this issue, we utilize density functional theory (DFT) to elucidate the fundamental oxidation mechanisms of SiN and SiC components by H_2O . Reaction rates were estimated with a combination of transition state theory and Arrhenius equation, enabling prediction of oxidation rates under various processing conditions. Additionally, H_2O diffusion within SiCN films was modeled, demonstrating a direct correlation between film density and oxidation kinetics. More importantly, the findings in this work can be applied in depositing SiOCN film as the oxidation mechanisms are predicted to be similar with other oxidation agents. Knowledge of these oxidation mechanisms enables precise control of the SiOCN film deposition process, facilitating component tunability.

11:45am **AP+AS+EL+EM+PS+TF-ThM-16 From Bulk Titanium Nitride to Small Molecule Inhibitors: a DFT Study Aiming Towards Area-Selective Atomic Layer Deposition**, **Lucas Lodeiro**, Universidad Tecnica Federico Santa Maria, Chile; **Marc J. M. Merks**, Eindhoven University of Technology, The Netherlands; **Dennis M. Hausmann**, **Rachel A. Nye de Castro**, LAM Research; **Adriaan J. M. Mackus**, Eindhoven University of Technology, The Netherlands; **Tania E. Sandoval**, Universidad Tecnica Federico Santa Maria, Chile

Titanium Nitride (TiN) is a hard and inert ceramic used as a protective coating, and in microelectronics for its metallic behavior. TiN thin films improve devices performance as conductive connection and diffusion barrier, and can be further functionalized to promote specific applications. Atomic Layer Deposition (ALD) enables precise TiN film deposition, with

temperature controlling crystal growth facet. However, achieving area-selective ALD (AS-ALD) on TiN is challenging, because the lack of information of surface groups present in deposited TiN, requiring reliable surface models to search for solutions for precursor selectivity and inhibition with Small Molecule Inhibitors (SMIs) at atomic scale.

This study uses Density Functional Theory (DFT) to examine TiN surface properties, crystal facets, and surface chemistry. It also explores the adsorption of various organic and inorganic precursor (Al, Si, Ti-based) and SMI (aryl, aldehyde, and nitrogen-based) molecules on TiN with the aim of studying their potential for AS-ALD processes with TiN as growth or non-growth area.

Our findings on crystal facets align with experimental data, showing the (001) facet is the most stable, followed by the (111) facet, which is observed at high deposition temperature.[1] The reactivity and functionalization strategies of these surfaces differ significantly. The (001) surface shows low reactivity (especially with H_2O , NH_3 , and H_2), resulting in bare surface sites.[2] Conversely, the (111) surface is reactive and can undergo hydrogenation, altering its electronic properties.

The differences in electronic surface properties significantly affect surface chemistry and the adsorption mechanism of the different molecules. The (001) surface exhibits metallic behavior, with strong interactions with various functional groups (for example, -1.8 eV for Benzaldehyde, BA), similar to copper surfaces.[3] In contrast, adsorption on the (111) surface is weaker and mainly dispersive (-0.8 eV for BA), highlighting the importance of the TiN film facet. Experimental findings show enhanced inhibition of BA and higher selectivity for low temperature deposited TiN, which could indicate the presence of the (001) surface, and a more stable inhibitor adsorption.

The key findings of this study offer valuable insights into surface reactivity and electronic properties to use TiN in AS-ALD process. Ultimately, this work aims to provide insights into controlling TiN deposition at the nanoscale, opening avenues for advanced microfabrication and surface engineering applications.

[1] *Met. Mater. Int.* 2001, 7, 621–625.

[2] *J. Phys. Chem. C* 2013, 117, 38, 19442–19453.

[3] *Chem. Mater.* 2025, 37, 1, 139–152.

12:00pm **AP+AS+EL+EM+PS+TF-ThM-17 Trimethylaluminum Reactivity on SiO₂ Surfaces at Cryogenic Temperatures – Implications for Al₂O₃ ALD**, **Leonhard Winter**, **Ravi Ranjan**, **Francisco Zaera**, University of California, Riverside

The atomic layer deposition (ALD) of aluminum oxide films on solid substrates using trimethylaluminum (TMA) and water is often considered a prototypical ALD process. Several investigations have attempted to understand the mechanistic details of this deposition by following the corresponding steps *in situ* under reaction conditions. To gain a more fundamental understanding, we have set out to study this system following a UHV surface-science approach, slowing down the reaction, decreasing the gas exposures and substrate temperature, and following the progress of the reactions using surface science techniques. We chose to study this chemistry on SiO₂ films grown *in situ* onto a Ta support because SiO₂ is one of the most common substrates in the microelectronics industry.

We investigated the adsorption and reaction of TMA with SiO₂ by using X-ray photoelectron spectroscopy (XPS) and temperature-programmed desorption (TPD). We found that TMA starts to react with the SiO₂ surface at ≈ 110 K, i.e. below the cryogenic temperatures required for multilayer condensation. This low-temperature chemistry appears to be complex, as multiple reaction pathways can be deduced from analysis of the TPD data. In addition to the expected product methane, we observed the formation of ethylene and heavier fragments, probably also containing Al. The complex behavior of TMA on SiO₂ is not limited to low temperatures, as the loss of alkyl groups continues over several hundred kelvins upon heating of the sample. Isothermal adsorption experiments show that at room temperature the TMA uptake is self-limiting with an initial sticking coefficient that is approximately 4–5 times smaller than at cryogenic temperatures, where multilayer growth occurs. To model ALD-type growth, we alternately dosed TMA and water at 200 K and followed the chemical composition of the surface with XPS. The results are in agreement with the expected ALD behavior, which shows that ALD growth is possible at these extremely low temperatures for the TMA/water system. The two precursors were also co-dosed in a CVD-type deposition, which results in the growth of multilayer films of aluminum oxide on the SiO₂ substrate. Surprisingly, the growth was observed to proceed faster at 200 K than at room temperature,

which we explain by a kinetic effect of prolonged residence times of the precursors at lower surface temperatures.

Plasma Science and Technology Room 201 ABCD W - Session PS1-ThM

Plasma Diagnostics

Moderators: **Thierry Chevolleau**, CEA-LETI, France, **Pingshan Luan**, TEL Technology Center America

8:00am PS1-ThM-1 Floating Probe-Based Plasma Potential Measurement in Low-Temperature Radio Frequency Inductively Coupled Plasma, Isak Lee, Chulhee Cho, Inho Seong, Wonnyoung Jeong, Minsu Choi, Byeongyeop Choi, Jami MD Ehsanul Haque, Seonghyun Seo, Woobeen Lee, Dongki Lee, Wonyun Park, Jinhyeok Jang, Shinjae You, Chungnam National University, Republic of Korea

Accurate measurement of plasma potential is essential for understanding sheath structures and particle dynamics. Among the available methods, the floating probe offers a simple approach but tends to underestimate the actual plasma potential due to electron flux from the plasma. To address this, we developed a modified floating probe that minimizes electron flux, enabling more accurate measurement of the plasma potential. The effectiveness of this technique was assessed by comparing the measured potentials with those obtained from a Langmuir probe. To evaluate their applicability, floating potentials were systematically measured under various conditions—including pressure, RF power, and probe configurations—and their variations were analyzed. As a result, we observed that reducing electron flux led the floating potential to approach the actual plasma potential. This study introduces a simplified and robust diagnostic method for plasma potential measurement, with high applicability to various plasma processing systems and low-temperature plasma research.

8:15am PS1-ThM-2 Global Model Enabled Quantitative Diagnosis of Reactive Species in a Plasma Chamber Using RGA, Seonghyun Seo, Wonnyoung Jeong, Chungnam National University, Republic of Korea; Sijun Kim, Laboratoire de Physique des Plasma (LPP)CNRS, Republic of Korea; Youngseok Lee, Chulhee Cho, Inho Seong, Minsu Choi, Byeongyeop Choi, Chungnam National University, Republic of Korea; Jami Md Ehsanul Haque, Chungnam National University, Bangladesh; Woobeen Lee, Isak Lee, Dongki Lee, Shinjae You, Chungnam National University, Republic of Korea

As plasma etching technologies become increasingly constrained with the advancement of high-aspect-ratio and high-precision patterning techniques such as atomic layer etching (ALE) and high aspect ratio contact (HARC) etching, the need for accurate control and quantitative analysis of reactive species within the process chamber has become increasingly important. Among the diagnostic tools used to analyze reactive species in the chamber, the residual gas analyzer (RGA) is widely adopted due to its accessibility, but its use has been largely limited to qualitative analysis.

This study proposes a diagnostic method to quantify radical densities by applying global modeling to RGA measurements. First, a Langmuir probe was inserted into the ionizer of the RGA to experimentally measure the electron density and electron energy distribution. These data were used as inputs for the global model to calculate electron-neutral collision rate coefficients for the radical species.

Then, to convert the measured RGA signals into absolute radical densities, we experimentally determined the mass-dependent transmission probability through a quadrupole mass filter, which reflects how the detection efficiency varies with species mass. By incorporating this transmission function along with previously obtained electron-related parameters, a global model was constructed to determine radical densities from the RGA signals.

To verify the reliability of the proposed method, it was compared with existing diagnostic approaches for quantifying radical species in plasma processes. Additional validation was carried out by evaluating the applicability of the global model under varying process conditions, including RF power and chamber pressure. This study demonstrates that reactive species in plasma environments can be quantitatively analyzed using the proposed RGA-based method.

8:30am PS1-ThM-3 Absolute Atomic Density Measurements in Hydrogen- and Oxygen-Containing Plasmas for Atomic-Scale Processing, Jente Wubs, Thomas van den Biggelaar, Marnix van Gurp, Erwin Kessels, Eindhoven University of Technology, Netherlands; Jordyn Polito, James Ellis, Harm Knoops, Oxford Instruments Plasma Technology, UK

INVITED

Hydrogen- and oxygen-containing plasmas are often used in atomic-scale processing technologies such as atomic layer deposition (ALD) and etching (ALE). Examples include the deposition of oxide layers and the etching of nitrides. To accelerate process development and optimization, physical analysis of the plasma is essential. In particular, measurements of key radicals – such as hydrogen and oxygen atoms – are required, as these radicals are known to affect on-wafer outcomes during ALD and ALE processes. Knowledge of their densities (and, ideally, their spatial and temporal distributions) is therefore of major importance, not only to understand the plasma chemistry pathways driving these processes, but also to identify relevant plasma regimes for achieving optimal processing conditions.

Available diagnostic techniques for measuring the densities of plasma radicals include probe-based methods and optical techniques, with the latter having the advantage of being non-invasive. A popular technique in both research and industry is optical emission spectroscopy. However, although this technique is experimentally relatively straightforward, analyzing emission spectra to obtain information on ground-state densities requires collisional-radiative models, which are only valid under specified conditions. Alternatively, ground-state densities can also be measured directly with absorption-based techniques, thus avoiding the need for modeling excitation processes. However, atomic absorption transitions from the ground-state to higher-energy states mostly lie in the vacuum ultraviolet part of the spectrum. The technical difficulties associated with this spectral region can be circumnavigated by using a technique called two-photon absorption laser induced fluorescence (TALIF), which does not require vacuum conditions and allows for measurements with high spatial and temporal resolution. However, TALIF is rather expensive and experimentally challenging, as it involves a bulky laser system and a complex calibration procedure. It is therefore less suited for monitoring atomic densities in industrial settings. Nevertheless, owing to the good accuracy and unparalleled spatial resolution, TALIF measurements are still of immense value when studying industrial plasmas, as they are necessary for the validation of e.g. probe-based methods and plasma models.

This contribution provides an overview of several plasma diagnostic techniques for detecting radicals relevant to ALD and ALE processes. Results on the densities of key plasma species in a commercial plasma source used for atomic-scale processing will be presented as well.

9:00am PS1-ThM-5 RF-Compensation-Free Langmuir Probe Technique via AC-Driven Biasing in a RF Plasma, Inho Seong, Chulhee Cho, Wonnyoung Jeong, Sijun Kim, Chungnam National University, Republic of Korea; Minsu Choi, Chungnam National University, Republic of Korea; Byeongyeop Choi, Chungnam National University, Republic of Korea; Ehsanul Haque Jami, Chungnam National University, Bangladesh; Seonghyun Seo, Woobeen Lee, Isak Lee, Dongki Lee, Chungnam National University, Republic of Korea; Shinjae You, Chungnam National University, Republic of Korea

Langmuir probe diagnostics in RF plasmas typically require filter to compensate for the RF fluctuations. This is commonly achieved by designing resonant filters that present high impedance at the fundamental frequency and its harmonics. However, fabricating such filters is often challenging due to the need for precise tuning and stability under plasma conditions, which can lead to increased system complexity. In this work, we present a novel method to perform Langmuir probe measurements without the need for conventional RF filters. By applying an AC-driven bias to the probe, we effectively suppress the influence of RF fluctuations, enabling direct plasma parameter measurements. We analyzed this novel technique and validated it through experiments, confirming the feasibility of simplified, filter-free probe diagnostics in a RF plasma.

9:15am PS1-ThM-6 Space and Phase-Resolved Ion Velocity Distribution Function Measurements in Electron Beam Generated E × B Plasma, Sung Hyun Son, Princeton University; Ivan Ramadanov, Princeton University Plasma Physics Lab; Nirbhav Chopra, Princeton University; Yevgeny Raitses, Princeton University Plasma Physics Lab

Electron beam (e-beam) generated plasmas with applied electric and magnetic ($E \times B$) fields are promising for applications that require efficient generation of ions and radicals in low-pressure environments [1]. We report spatially and phase-resolved measurements of the ion velocity distribution function (IVDF) in this plasma source using a planar laser-induced

fluorescence (PLIF) system. A continuous-wave tunable diode laser produces a laser sheet that irradiates the plasma, and the resulting fluorescence is captured by an intensified CCD (ICCD) camera. Fluorescence images recorded at varying laser wavelengths are converted into two-dimensional IVDFs using the Doppler shift principle [2]. The PLIF measurements are validated against a conventional single-point laser-induced fluorescence (LIF) method using photomultiplier tube (PMT)-based detection at various positions. The phase-resolving capability of the system is tested by oscillating the plasma between two nominal operating modes with distinct density profiles, with the ICCD camera triggered by the externally driven plasma oscillation. The resulting oscillations in fluorescence intensity show good agreement with plasma density variations measured by electrostatic probes, demonstrating the system's ability to resolve phase-dependent dynamics. The measured IVDFs reveal several signatures of ion dynamics in this plasma source that could influence its material processing characteristics. In particular, radially outflowing ions and anomalous ion heating in the plasma periphery, both anticipated by theoretical studies and potentially detrimental to gentle plasma processing [3], are observed and reported.

[1] Zhao F, Raitses Y, Yang X, Tan A, Tully C.G. 2021 High hydrogen coverage on graphene via low temperature plasma with applied magnetic field *Carbon* 177: 244-251

[2] Severn G D, Edrich D A and McWilliams R 1998 Argon ion laser-induced fluorescence with diode lasers *Rev. Sci. Instrum.* 69 10–5

[3] Chopra N S, Romadanov I and Raitses Y 2024 Production of warm ions in electron beam generated $E \times B$ plasma *Appl. Phys. Lett.* 124064101

9:30am **PS1-ThM-7 Short Duty Cycle Pulsing of an RF Driven ICP with Electronegative Gases**, *Banks Peete, Carl Smith*, North Carolina State University; *James Prager, Paul Melnik, Tim Ziemba*, Eagle Harbor Technologies; *Sung-Young Yoon, Meehyun Lim, Sungyeol Kim*, Samsung Electronics, Republic of Korea; *John Mattingly, Steve Shannon*, North Carolina State University

Many common gases used for plasma enhanced processes in semiconductor manufacturing have electronegative properties; the gas molecules will attach free electrons in a plasma to form negatively charged ions. These gases are a vital presence in etching processes because of their ability to form certain reactive species. However, electron attachment can form plasma instabilities that vary in amplitude and frequency based upon the power delivery design, power density, and gas composition. These instabilities disrupt power delivery, leading to challenges in consistency for industrial applications. Power delivery networks that do not rely on traditional impedance matching have been studied previously to demonstrate expanded process capabilities for pulsed RF power delivery, most notably through reduced power delivery latency and more rapid electron-ion pair production. This work expands the study to evaluate the performance of a matchless RF power delivery network with regard to electron-ion pair production, plus power delivery latency and stability when used with an electronegative plasma. The ability to quickly apply RF power with minimal delay, enabled by the matchless pulser, allows the RF to be turned on and off before the instabilities can fully manifest while still producing a controllable peak electron density over a short pulse cycle time. Thus, the plasma avoids the onset of the instability in electron density and temperature that is characteristic of electron attachment instabilities in electronegative plasmas by achieving the desired peak density on a time scale faster than the onset of the instability. This can expand the stable operating space for industrial plasmas reducing the reliance on very specific gas mixture, pressure, and power parameters where the instability does not occur.

This work is supported by a grant from the Samsung Mechatronics Research Division, Suwon, Republic of Korea.

9:45am **PS1-ThM-8 Probing Microwave-Driven Plasmas: Impact of N_2 Addition in Ar/N_2 Plasma**, *Nafisa Tabassum*, North Carolina State University; *Abdullah Zafar, Timothy Chen, Kelvin Chan*, Applied Materials; *Steven Shannon*, North Carolina State University

A microwave-driven plasma operating at 2.45 GHz is investigated by means of optical emission spectroscopy, laser absorption spectroscopy, laser induced fluorescence, probe diagnostics, and plasma simulation package Zapus. A mixture of Ar/N_2 is used as the operational gas with N_2 partial pressure varied from 0 % to 25 % of total gas pressure. The effect of N_2 partial pressure, gas pressure, and delivered power density are investigated in the range of 70 mTorr - 1 Torr and 0.25-1.25 W/cm³. Electron density, electron temperature and plasma potential were measured using a single Langmuir probe. Imaging of the plasma using an ICCD camera was used to

estimate the physical extent of the plasma. Relative concentrations of molecular nitrogen N_2 , ionized molecular nitrogen N_2^+ and atomic nitrogen N were obtained through optical emission actinometry as a function of pressure and delivered power density. The following lines are used in this study: N_2 : $C^3\Pi_u \rightarrow B^3\Pi_g$ at $\lambda = 337.1$ nm, N_2^+ : $B^2\Sigma_u^+ \rightarrow X^2\Sigma_g^+$ at $\lambda = 391.0$ nm, N: $3p^4S_{3/2} \rightarrow 3s^4P_{5/2}$ at $\lambda = 746.8$ nm, and Ar: $2p_1 \rightarrow 1s_2$ at $\lambda = 750.4$ nm. The transition between the under-dense and over-dense operating regimes, influenced by variations in the delivered power density, gas pressure, and N_2 partial pressure has been mapped to these plasma generated species. This study identifies hysteresis effects during changes in delivered power densities and pressure near the critical power, P_c , for transition from the under-dense to over-dense condition. In particular, the critical power required for this transition decreases with increasing pressure. This hysteresis behavior is further confirmed through observations of plasma diameter variations under different pressure and power density conditions. The influence of the partial pressure of N_2 in $Ar - N_2$ plasma on the mode transition and hysteresis is investigated. This study explores how plasma-generated species form, their roles in ionization pathways within a multi-species gas mixture, and how these factors affect the transition from under-dense to over-dense regimes. These dynamics, in turn, influence the critical power required for the transition and the spatial distribution of the plasma-generated species.

Plasma Science and Technology Room 201 ABCD W - Session PS2-ThM

Plasma Sources

Moderator: Necip Uner, Middle East Technical University

11:00am **PS2-ThM-13 Controlled Electron-Enhanced Silicon Etching with H_2 Background Gas and Positive Sample Voltage**, *Sumaira Yasmeen, Andrew Cavanagh*, University of Colorado at Boulder; *Harsono Simka, Samsung Electronics; Steven George*, University of Colorado at Boulder

Controlled electron-enhanced silicon etching can be achieved with H_2 background gas and positive sample voltage. Electrons impinging on the silicon surface at normal incidence at currents of ≥ 200 mA over surface areas of ~ 4 cm². The electron energy was ~ 140 -240 eV defined by the grid bias on the hollow cathode plasma electron source and positive sample voltages. The H_2 pressures were < 3 mTorr. The silicon etching for Si(100) and a-Si at room temperature was measured using in situ spectroscopic ellipsometry. The etched silicon thickness was linear versus time during electron-enhanced etching. The etch rates increased progressively with larger positive sample voltages (Figure 1). Si(100) etched slower than a-Si. For example, the etch rates were ~ 2.6 Å/min for crystalline Si(100) and 9.9 Å/min for a-Si under the same conditions at an incident electron energy of 140 eV with a positive sample voltage of +90V.

Without the positive sample voltage, the silicon etch rates were negligible. In addition, electron-enhanced Si etching was not accomplished using a D_2 background gas instead of a H_2 background gas. These results support the proposed mechanism for electron-enhanced Si etching, where H_2 produces H^- via dissociative electron attachment (DEA) according to $H_2 + e^- \rightarrow H_2^- \rightarrow H + H^-$. The positive voltage on the sample stage then pulls the H^- negative ions to the silicon sample to react with silicon to produce SiH_4 as an etch product (Figure 2). The low-energy electrons required for DEA are secondary electrons produced by the primary electrons impinging on the silicon surface.

The energy of secondary electrons from silicon peaks at ~ 2 -3 eV and drops off rapidly at higher energies approaching 10 eV. The peak of the DEA cross section for H_2 is 3.75 eV. In comparison, the peak of the DEA cross section for D_2 is 14.0 eV. The D_2 background gas may not be effective for silicon etching because D^- is not produced by DEA, as the secondary electron energy from silicon is too low. These results demonstrate a new mechanism for controlled electron-enhanced silicon etching based on H_2 DEA from secondary electrons and H^- attraction to the positive sample voltage on the silicon sample.

11:15am **PS2-ThM-14 Investigation of Temporal, Spatial and Angular Evolution of High-Power Impulse Magnetron Sputtering with Positive Cathode Reversal**, *Tag Choi, Zachary Jeckell, Sam Pickholtz, Matt Egly, Matt Salek, Ricky Pickering, Aaron Hackett, Dren Qerimi, David Ruzic*, University of Illinois at Urbana-Champaign

High-Power Impulse Magnetron Sputtering (HiPIMS) is a Physical Vapor Deposition (PVD) technique that delivers sub-microsecond high power pulses, enhancing ionization rate, ion energy and its directionality.

Thursday Morning, September 25, 2025

Furthermore, a positive cathode reversal feature has been added to enhance the deposition rate and control the ion energy. These HiPIMS capabilities contribute to notable improvements in film quality, including higher density, stronger substrate adhesion, and enhanced step coverage, making HiPIMS with positive cathode reversal well-suited for advanced thin-film applications. Among the many challenges in the semiconductor industry, achieving uniform and dense thin films remains a critical focus. Addressing this requires a deeper understanding of the underlying plasma behavior during deposition. Thus, this study utilizes a Plasma Sampling Mass Spectrometer (PSM) to investigate the temporal, spatial, and angular evolution of titanium HiPIMS plasma with positive cathode reversal. A custom-built, high-vacuum compatible magnetron was mounted on both linear and rotational actuators, enabling detailed exploration of ion behavior across different positions and angles. The system allows for 40–300 mm axial translation, 90 mm azimuthal movement, and up to 90° rotation. Key HiPIMS parameters—such as main pulse duration, peak current, delay time between the main and positive kick pulses, kick pulse length, and kick voltage—were systematically varied to assess their individual impact on plasma characteristics. Additionally, the study examines the controllability between gas and metal ions, exploring its potential for process optimization and application-specific tailoring.

11:30am **PS2-ThM-15 University-Scale Extreme-Ultraviolet Lithography Source**, Jan Uhlig, Max Miles, Dren Qerimi, David Ruzic, University of Illinois at Urbana Champaign

To elevate our laboratory's research capabilities in Extreme-Ultraviolet (EUV) photoresist development, we have engineered a university-scale EUV light source designed for lithography applications at a fraction of the cost of commercial EUV tools. This innovative system leverages a Neodymium-doped yttrium aluminum garnet (Nd:YAG) Laser-Produced Plasma (LPP) with a swappable Tin (Sn)-based target to generate EUV radiation, offering an economical alternative to the high-cost infrastructure typically required for EUV lithography. Departing from an earlier dual EUV Multilayer Mirror (MLM) configuration, the current setup employs direct EUV exposure facilitated by a 150 nm Zirconium (Zr)-based transmission filter to isolate the EUV spectrum with high efficiency. A broad-spectrum photodiode detector, tailored to EUV wavelengths, provides accurate dosimetry, and its measurements are corroborated through successful exposure of EUV-sensitive photoresist-coated wafers. Experimental investigations have explored a range of target materials, including pure Sn and Sn-doped ceramics (typically 5 at%), utilizing Spectraflux 100B (Lithium Metaborate/Lithium Tetraborate 80/20) as a foundational component. Optical Emission Spectroscopy (OES) serves as an auxiliary diagnostic tool, enabling real-time monitoring of the LPP characteristics. Optimization efforts have focused on critical parameters—laser power, beam focus, operating pressure, and target composition—to achieve precise dose control and maximize EUV output while simultaneously addressing debris mitigation, a persistent challenge in LPP systems. The results demonstrate reliable photoresist exposure with adjustable EUV dosage, positioning this system as a cost-effective yet powerful platform for academic research. Compared to multimillion-dollar commercial EUV tools, this setup provides an accessible means to explore novel EUV photoresist modifications, with ongoing projects targeting enhancements in line-edge roughness and reductions in the minimum dose required for full development. This affordable, university-scale tool thus bridges a critical gap, enabling advanced EUV lithography studies without the prohibitive expense of industrial-grade equipment.

11:45am **PS2-ThM-16 Understanding the Plasma in Dielectric Barrier Discharges for Plasma-Enhanced Spatial Atomic Layer Deposition**, Ralph Houben, Antoine Salden, Jente Wubs, Richard Engeln, Erwin Kessels, Julian Held, Bart Macco, Eindhoven University of Technology, Netherlands

Plasma-enhanced atomic layer deposition (PE-ALD) has enriched the ALD field, with the non-equilibrium nature of the plasma allowing for the synthesis of novel materials, tuning of material properties, and deposition at low temperatures. Pivotal to advancing the field of PEALD has been the detailed understanding of the plasma chemistry, including the role of reactive species and ions in driving surface reactions.

However, many emerging application in green technologies, including photovoltaics, batteries, and catalysis, require high-throughput, large-area deposition methods. Batch (thermal) ALD has already proven itself in the field of passivation layers for solar cells, yet plasmas are not easily integrated into such batch ALD systems. Spatial ALD (SALD) offers a route to incorporate plasmas using dielectric barrier discharges (DBDs), operating at atmospheric pressure and over large areas. Yet, the properties of DBDs and

their influence on ALD deposition remain far less understood compared to the well-studied conventional PEALD plasmas.

We demonstrate how the DBD is implemented in the SALD tool used for deposition. The SALD setup consists of a head with multiple parallel slits through which precursors and co-reactants flow, separated by inert gas. The DBD plasma is applied over one of these gas channels, enabling plasma-enhanced surface reactions in a spatially separated, continuous process. This approach allows us to combine the scalability of SALD with the enhanced reactivity of plasma species.

We furthermore present a systematic study of a DBD source that is currently being integrated on our spatial ALD tool. Using optical emission spectroscopy, we investigate the gas temperature in the afterglow of the N₂/O₂ plasma, finding temperatures between 310 and 400 K depending on input power, which is compatible with ALD processes. Additionally, broadband absorption spectroscopy is used to quantify reactive species including O₃ and NO_x radicals, as function of N₂/O₂ ratio and input power, finding ozone densities of 10¹⁵ cm⁻³. This indicates that the reactive species flux can be finely tuned via gas composition. At low O₂ dilution, we have indications that atomic O can become an important radical species. Current work focuses on the influence of these different radical regimes on the deposition processes on our spatial ALD tool for various materials, and their properties.

To conclude, our work contributes to bridging the gap between DBD plasma physics and ALD chemistry for plasma-enhanced spatial ALD, providing a pathway toward optimizing plasma conditions for scalable, atmospheric-pressure ALD processes aimed at clean energy applications.

Atomic Scale Processing Mini-Symposium Room 206 A W - Session AP+PS+TF-ThA

Emerging Applications for Atomic Scale Processing (ALD/ALE) including Precursors and Surface Reactions

Moderator: Robert Bruce, IBM Research, T. J. Watson Research Center

2:15pm **AP+PS+TF-ThA-1 ALD Thin Films for Protecting Limestone Cultural Heritage**, *Gillian Boyce*, *Suveena Sreenilayam*, University of Maryland, College Park; *Eleonora Balliana*, *Elisabetta Zendri*, Università Ca' Foscari Venezia, Italy; *Raymond Phaneuf*, University of Maryland, College Park

From natural erosion to pollution-accelerated decay, stone cultural heritage deteriorates constantly through interactions with the environment. Common protective treatments such as acrylic polymers are generally prone to degradation and loss of performance, and they are often limited in their ability to achieve uniform and conformal coverage across a stone's topographical features. In this work, we report on the results of investigations of atomic layer deposited (ALD) amorphous alumina thin films for the protection of calcium carbonate substrates of a wide range of porosity against acid-based dissolution. The protective effects of the ALD coatings were investigated by aqueous acid immersion. The solution pH was tracked over time for a constant volume of acetic acid solution with an initial pH of 4 with the stone samples immersed. We find the protective effect of ALD alumina coatings is extremely promising, with 90 nm thick coatings slowing the average rate of pH evolution significantly, by between one and two orders of magnitude, depending on the porosity of the substrate. The eventual failure of the ALD coatings during immersion was also investigated, with the development of pits on the substrates, whose area fraction correlates to the changing pH of the acid solution during immersion. The variation of the protective action of the films with thickness is consistent with kinetics which are limited by diffusion within the pits, rather than through the films. Our findings point to the dominant role of defects in the thin films in their eventual failure

2:30pm **AP+PS+TF-ThA-2 Atomic Layer Deposition on Ceramic Nanopowders for Precisely Engineered Microstructure of Sintered Ceramics**, *Eric Bissell*, *Alexandros Kostogiannes*, *Steve Lass*, *Anna Zachariou*, *Brian Butkus*, *Luis Tomar*, *Terrick Mcnealy-James*, *Ayelen Mora*, *Blaine Mauri-Newell*, University of Central Florida; *Nicholas Rudawski*, University of Florida, Gainesville; *Romain Gaume*, *Parag Banerjee*, University of Central Florida

In this work, we have utilized the conformal nature and monolayer control of growth of ALD films to develop ≤ 10 nm, ultrathin diffusion barriers on the surfaces of ceramic nanoparticles. The barrier layer restricts grain growth during sintering leading to formation of bulk, nanocrystalline ceramics which demonstrate unique properties such as superior hardness and optical transparency, otherwise not achievable using traditional powder preparation and sintering steps.

Zinc oxide (ZnO) nanoparticles of 60 nm nominal diameter were coated with 1 or 10 nm of Al₂O₃ in a custom-built, rotary ALD powder reactor. In situ mass spectrometry was used to end point the half-reaction pulse times. The powder was subsequently mixed at a 1:1 mass ratio with uncoated ZnO nanoparticles where the uncoated ZnO served as the 'control' sample undergoing the exact thermal and pressure cycling as the coated regions. The powder mixtures were subsequently compacted and hot pressed at 850 °C under uniaxial loading of 150 MPa. The sintered ceramics reveal that the 1nm and 10nm 'shell' Al₂O₃ layers effectively restrict grain size of the ZnO to 89 ± 23 nm and 55 ± 7 nm respectively, whereas the uncoated regions grow large polycrystalline grains of 601 ± 104 nm and 717 ± 80 nm respectively. The crystal structure analysis reveals ZnO in its thermodynamically stable wurtzite phase with no evidence of secondary phase formation. This study demonstrates the broad applicability of ALD based coating technology to the field of ceramics for fine microstructural control and precise tunability of bulk properties.

2:45pm **AP+PS+TF-ThA-3 Hot-Wire-Assisted Atomic Layer Deposition of Transition Metals**, *Kyeongmin Min*, *Han-Bo-Ram Lee*, Incheon National University, Republic of Korea

To replace conventional Cu interconnects, atomic layer deposition (ALD) of low figure-of-merit (FOM) materials such as cobalt (Co) and nickel (Ni) is crucial. While noble metals have been extensively studied as alternative interconnect materials due to their excellent performance, the high cost necessitates the development of low cost materials with superior

properties. However, existing Co and Ni ALD processes inevitably require plasma to achieve high purity, leading to inherent limitations such as poor step coverage due to radical recombination and unavoidable damage to 3D structures caused by energetic ions and photons. In this study, we studied transition metal ALD processes using a hot-wire-activated counter reactant, enabling the deposition of high-purity films without generating energetic ions or photons. NH_x radicals were generated by exposing NH₃ counter reactant gas to a filament heated over 1300 °C. Due to the high thermal energy of the filament, NH₃ gas molecules dissociated into high energy radicals, which played a crucial role as reactants in the transition metal ALD processes. The concentration of NH_x radicals was studied as a function of the hot wire temperatures and correlated with the physical properties of films. The purity of transition metal films was analyzed using X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES). Based on the results of this study, we believe that the hot-wire-assisted ALD process can be widely utilized in various applications where overcoming the limitations of conventional plasma ALD is essential.

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3:00pm **AP+PS+TF-ThA-4 Ni Thin Film Deposition Using Hot Wire ALD and Non-Halogen Precursor**, *Mruthunjaya Uddi*, *Mike Denchy*, *Prawal Agarwal*, *Josh Kintzer*, *Patryk Radyjowski*, Advanced Cooling Technologies Inc.

Scale up of pure phase nickel (Ni) thin film deposition process for various applications of catalysis, microelectronics, chemical sensors, and MEMS, especially, using environmentally friendly non-halogen precursors is challenging. ALD is a variation of Chemical Vapor Deposition (CVD), with the complete metal deposition reaction broken into two half steps. Since each half-step saturates at a single atomic layer, a very precise control over deposition can be achieved. Although slower than CVD deposition rates, ALD can enable precise, uniform, conformal coating of Ni thin films. Recently, we assembled an automated Hot Wire Atomic Layer Deposition (HW-ALD) reactor and demonstrated Ni thin film deposition using a non-halogen precursor nickelocene and NH₃. The hot wire implementation enabled the non-halogen chemistry pathway. The details of reactor design, operation parameters and characterization of the Ni thin film deposited will be presented. Future experiments will involve large area (> 15 cm diameter) substrate coating with Ni thin films and the uniformity of distribution will be studied.

3:15pm **AP+PS+TF-ThA-5 Pyroelectric Calorimetry for ALD**, *Ashley Bielinski*, Argonne National Laboratory

A deeper understanding of the self-limiting surface reactions that make up and ALD processes is vital for the development of many emerging applications such as area and site selective ALD processes that rely on chemical differentiation between a range of surface sites. Natural variation and defects in real surfaces necessitate in situ measurements of these surface reactions in order to develop a complete picture of the process. These in situ measurements can be combined with computational results on simplified model surfaces to help understand not only the single most favorable reaction pathways but also changes in the reactions as surfaces dynamically approach saturation and reactions on a realistic range of surface conditions.

Pyroelectric calorimetry can be used to quantitatively measure the heat evolved during an ALD surface reaction with high time resolution within a single saturating precursor reaction. This approach has been used to measure the reaction enthalpy of various ALD precursor reactions during the deposition of Al₂O₃, ZrO₂, and MgO. Analysis of the heat generation rate profiles of these processes in combination with techniques such as in situ spectroscopic ellipsometry and quartz crystal microgravimetry have provided insight into properties including multi-step reaction mechanisms and the driving role of entropy in certain reaction mechanisms. Recent hardware developments further enable measurements of precursor delivery and reaction kinetics. Knowledge of the mechanisms, thermodynamics, and kinetics of these reactions will guide the development of future ALD processes and provide the necessary parameters for the development of more complex and accurate computational models.

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3:30pm AP+PS+TF-ThA-6 Fabrication of Atomically-Precise Nanoimprint Masks by STM Lithography, James Owen¹, Ehud Fuchs, John Randall, Zyvex Labs

The Semiconductor industry is struggling to continue to follow Moore's Law. For both technical and economic reasons, it is likely that the ASML High-NA Extreme Ultraviolet Lithography (EUV) tools will be the last photolithography technology to push to higher resolutions. Simultaneously, E-Beam Lithography (EBL) mask writers, while improving throughput by going highly parallel, are also very near the end of resolution improvements. The industry does not appear to expect any significant downscaling of devices beyond what will be possible with the ASML High-NA EUV tool which has a resolution of 8 nm.

The DOE Advanced Materials and Manufacturing Technologies Office (AMMTO) sponsored Semiconductor Industry Energy Efficiency Scaling (EES2) roadmap has identified EUV as a significant contributor to the energy budget of advanced digital electronics. Strikingly, EUV is so inefficient that only about 0.04% of the beam energy actually affects the resist. The EES2 roadmap proposes that replacing EUV with Nanoimprint lithography (NIL) would be a way to improve the energy efficiency of semiconductor manufacturing. NIL offers equal and better resolution and precision than EUV, with up to 90% lower energy costs, resulting in lower costs of production. However, NIL uses a mold of the pattern to be printed on the wafer as a mask and the best resolution of the current EBL mask writers is 15nm. Therefore, a mask writing technology with better resolution than EBL is required; it must provide resolution at least as good as the High NA EUV tool's 8nm to be widely adopted.

We describe a pathway towards unprecedented resolution in nanoimprint mask fabrication. Ultrahigh-precision NIL templates are made by writing sub-nm-precision patterns on Si(001) using Scanning Tunneling Microscope (STM) lithography followed by selective growth via atomic layer deposition of a hard mask such as TiO₂, which is then used as an etch mask for Reactive Ion Etching to form a Si template, replicating the STM pattern. This template would then be transferred into a quartz template using existing step and flash NIL processes which will then be used to pattern devices on the die or wafer scale. We show that sub-10 nm feature sizes and full-pitch gratings with feature radius of curvature down to 1.5 nm in the lateral dimension are achievable, although the throughput is currently much too slow to be industrially feasible at the moment. This process therefore addresses the EES2 goal of improving the energy efficiency during manufacturing of digital electronics.

3:45pm AP+PS+TF-ThA-7 Three-In-One Isolation New Integration Solution for Monolithic CFET, Junjie Li, Longrui Xia, Institute of Microelectronics of the Chinese Academy of Sciences, China; Mingmei Wang, Lam Research Corporation

Introduction: Monolithic complementary transistors (CFET) will replace gate all around field-effect transistor (GAA FET) in technology nodes below 1nm [1]. CFET stacks N-type transistors on top of P-type transistors to reduce footprint and increase transistor density. Therefore, it is important to isolate the gate and source drain, as well as the gate of the upper and lower transistors. The current publicly available solution is to isolate the gate and source drain of the outer wall and inner wall, and isolate the gate of the upper and lower transistors with an intermediate isolation layer (MDI). This is achieved through three process steps, requiring at least three atomic layer deposition and atomic layer etching[2] (figure 1 of supplemental material). In order to simplify the process and reduce its difficulty, this article proposes a new process integration scheme that combines the above three steps into one deposition step and one etching step to achieve.

Results and Discussion :

Conclusion: We conducted a detailed comparison between the publicly available CFET process flow and the new process flow proposed in this article, as shown in Figure 2 f supplemental material, and ultimately successfully implemented a new three-in-one integrated solution of inner and outer side spacers and MDI. And demonstrated the results of key intermediate steps such as concave dummy gate and CH_x dummy gate self-aligned etching source drain.

Reference:

- [1] C. Cavalcante, VLSI, (2025).
- [2] T.Lill, VLSI (2025).

4:00pm AP+PS+TF-ThA-8 New Silicon-containing Precursors for Metal Silicide Films, Sean Barry, Dexter Dimova, Carleton University, Canada

Group 3 metals, particularly scandium and yttrium, offer tunable electropositivity when incorporated in thin films. They are attractive for next-generation microelectronic applications, including silicide and germanide thin films in gate-all-around FET architectures. However, suitable precursors for vapor phase deposition of lanthanide films remain scarce. In this work, we explore a new family of geminal diamidosilane (gDAS) ligands, designed for both homoleptic and heteroleptic coordination to Group 3 centers. These ligands provide modular steric control and enhanced volatility, allowing for the design of thermally stable precursors.

We report the synthesis and characterization of several Sc and Y complexes with methyl- and tert-butyl-substituted gDAS ligands. Thermogravimetric and isothermal analyses reveal decomposition pathways involving γ -hydride elimination, which is critical for understanding volatility and thermal stability in thin film deposition. Notably, Y(gDAS)₃ precursors exhibit promising volatility, while extended thermolysis suggests silicide incorporation may be feasible under atomic layer deposition conditions. We also demonstrate preliminary hydrogenation and dehydrocoupling strategies to prepare silicon-containing intermediates compatible with gDAS ligand frameworks. This presentation will show the groundwork for tailored ligand design strategies that enable selective, low-temperature deposition of lanthanide-silicon films.

4:15pm AP+PS+TF-ThA-9 Conversion-free Atomic Layer Etching of ZnO Using Hydrofluoric Acid and Trimethylgallium for Reduced Residues, Taewook Nam, Sejong University, Republic of Korea; Steven George, University of Colorado Boulder

Thermal atomic layer etching (ALE) is a crucial technique for advanced semiconductor manufacturing, offering precise material removal with sub-nanometer control. While various etching mechanisms exist, including the widely used fluorination-ligand exchange reaction, some materials are etched via a "conversion" mechanism where the original material is converted into a different compound before being removed. This conversion-based ALE, as seen in zinc oxide (ZnO) etching with hydrofluoric acid (HF) and trimethylaluminum (TMA), can leave undesirable residues of the converted material, Al₂O₃, on the surface, which is detrimental for fabricating high-performance, sub-nanometer scale devices.

In this study, we present a novel, conversion-free thermal ALE process for ZnO using HF and trimethylgallium (TMG) as precursors. This alternative chemistry was investigated using a suite of characterization techniques, including quartz crystal microbalance (QCM) and quadrupole mass spectrometry (QMS). ZnO films were initially grown via atomic layer deposition (ALD) using diethylzinc (DEZ) and water at 100°C. QCM measurements during the ALE process showed self-limiting mass gain during HF exposure and mass loss during TMG exposure, confirming the characteristic digital nature of the process. The etch rate was found to increase with temperature, reaching 3.82 Å/cycle at 300°C.

A significant advantage of the HF-TMG process is its ability to etch at temperatures as low as 30 °C, which is dramatically lower than the ≥ 240 °C required for the conventional HF-TMA process. This difference in temperature is attributed to the distinct reaction mechanisms of the metal precursors. While TMA exposure converts ZnO to Al₂O₃, enabling the subsequent unwanted deposition of AlF₃ during HF exposure, TMG exposure results in mass gain from precursor adsorption without conversion. The absence of this competing ALD reaction allows for effective, low-temperature ALE with HF-TMG. QMS analysis further supports this, showing no evidence of conversion and confirming a fluorination-ligand exchange mechanism.

The conversion-free nature of the HF-TMG process also leads to a notable reduction in residual contamination on the etched surface. X-ray photoelectron spectroscopy (XPS) confirms that the HF-TMA process leaves significant concentrations of both F (8.6 at.%) and Al (8.9 at.%) on the surface after ALE. In contrast, the HF-TMG process results in much lower residual concentrations of both F (1.1 at.%) and Ga (2.4 at.%).

Thursday Afternoon, September 25, 2025

Plasma Science and Technology

Room 201 ABCD W - Session PS+AIML-ThA

Plasma Modelling AI/ML

Moderators: Kenji Ishikawa, Nagoya University, Japan, Angelique Raley, TEL Technology Center, America, LLC

2:15pm PS+AIML-ThA-1 Machine Learning for Low Temperature Plasma Applications, Abhishek Verma, Kallol Bera, Shahid Rauf, Applied Materials, Inc. INVITED

Low temperature plasmas are used for numerous depositions and etch applications in the semiconductor industry. The field is rapidly advancing driven by volumes of multimodal and complex spatiotemporal data from both experiments and simulations. Machine learning in combination with plasma modeling and simulation offers a wealth of techniques that could play key role in plasma source discovery, design and decision making. These techniques can also augment domain knowledge for plasma reactor control and process development. In this talk, we present our work on machine learning applications to modeling, control, and optimization of plasma chambers. To overcome the challenge of high computational cost associated with high fidelity plasma models for rapid and many-query analyses, we present a deep learning based non-linear surrogate modeling method. Our numerical experiments on capacitively coupled plasmas show that deep learning-based model can learn an efficient latent space representation of spatiotemporal features of plasma characteristics. Moreover, we extended this approach with physics informed neural networks to improve predictive accuracy and generalization while being data efficient. Physics informed approaches could also effectively incorporate expert knowledge while learning physics implicitly. Furthermore, we present application of regression methods for circuit estimation of collisional sheath in moderate pressure capacitively couple plasmas. The novel sheath model which includes collisional effects, ion current responses to sheath voltage and harmonics based resistive elements, builds on parametric flexibility using machine learning while maintaining interpretability. The talk outlines machine learning methodologies for modeling, optimizing, and controlling plasmas for semiconductor applications.

2:45pm PS+AIML-ThA-3 Contour-Based Objectives for Robust Etch Model Selection, Chad M. Huard, Prem Panneerchelvam, Shuo Huang, KLA; Lewis Hill, Janet Hopkins, KLA UK; Mark D. Smith, KLA

As device scaling increasingly relies on 3D rather than CD scaling, etch has become a critical and challenging step, often limiting further scaling. The demand for high-quality, predictive etch models is growing, yet our understanding of surface mechanisms during dry etching remains limited. Techniques like XPS, SIMS, and AES provide clues to surface reactions, but the pathways are not immediately clear. First-principles computational methods such as DFT, quantum MD, and classical MD offer insights but are constrained by computational resources and turnaround times. We present a Monte Carlo profile model that bridges the gap between first-principles and empirical models by using simplified chemistry mechanisms calibrated with experimental data. Traditional models often rely on 'best-effort' mechanisms, risking calibration issues due to high dimensionality or model errors due to omission of critical pathways. We propose a unified method for evaluating etch mechanisms using rigorous contour-based objectives, which maximizes entitlement from metrology data and results in better model development/selection compared to gauge-based metrics. This approach identifies the simplest model that fits the data, addresses degeneracy in models and calibration objectives, and enhances model predictiveness.

3:00pm PS+AIML-ThA-4 NAND Pillar Etch: Plasma and Feature Profile Modeling in Dry Etch Process, Harutyun Melikyan, Ebony Mays, NAND Pathfinding - Micron Technologies; Ali Bhuiyan, Sumeet Pandey, Advanced Modeling - Micron Technologies; Jagannath Mahapatra, Micron Technologies, USA

In this work we developed a model to study the Feature Profile Modeling (FPM) in the dry etch plasma process for NAND pillar etch. The model developed takes in process parameters, that is process knobs such as temperature, pressure, flowrates, Power, Frequency, Voltage as inputs. The output from the model is Feature profile information such as Etch rate, Etch Depth, Variation of CD with height, Twisting, Ellipticity, Necking (HM), Bowing (ONO) etc. This methodology makes possible the ability to correlate process knobs on an equipment directly to the feature profile. This can enable us to get a detailed sensitivity analysis of feature profile with respect to process knob on the equipment (like constructing a sort of digital

twin for that equipment). In addition, the feature profile (for HAR) for the future nodes can be inferred from process knobs and recipe information even before running the experiments.

3:15pm PS+AIML-ThA-5 Machine Learning of Simulated Nanosecond UV Laser Ablation Plumes, Jacob Paiste, University of Alabama at Birmingham; Sumner Harris, Oak Ridge National Laboratory; Shiva Gupta, University of Alabama at Birmingham; Eric Remington, Samford University; Robert Arslanbekov, CFDR Research Corporation; Renato Camata, University of Alabama at Birmingham

Laser-generated plasmas are a rich laboratory of complex spatiotemporal phenomena emerging from coupled thermodynamic, electromagnetic, and quantum mechanical processes. The strength of laser-solid and laser-plasma interactions can vary over multiple orders of magnitude while gradients of density, temperature, and flow velocity give rise to shocks, instabilities, and turbulence in multiphase flows. Deep learning can be used to encode these complex spatiotemporal dynamics to discover correlations between the conditions under which a laser-generated plasma is produced—including the wide chemical and thermophysical diversity of ablation targets—and the resulting plasma flows. Predictive models can then be built to infer the fundamental properties of irradiated solids and plasmas, enabling a new experimental modality for measuring material properties like thermal conductivity or critical temperature. However, no databases of experimental or simulated laser-generated plasmas currently exist, so proof-of-concept for the efficacy of deep learning for this task is difficult to obtain.

In this work, we carry out a deep learning study on synthetic laser-generated plasma data. The synthetic data sets are produced using a combined laser ablation-fluid dynamics simulation based on the Hertz-Knudsen model, including phase explosion when a target temperature exceeds the thermodynamic critical temperature. The model is implemented on an open-source Adaptive Cartesian Mesh framework that enables laser ablation plume simulations out to centimeter distances over tens of microseconds for any elemental material with well-defined thermophysical parameters.

We generate a training dataset by simulating UV nanosecond pulsed laser ablation of elemental targets of Be, B, Na, Mg, Al, Sc, Ti, V, Fe, Co, Cu, Zn, Rb, Cs, Ta, W, and Pt with systematic variation of laser fluence (1–10 J/cm²) and laser spot area (0.8–13 mm²). We use (2+1)D convolutional neural networks (CNNs) to encode spatiotemporal plume dynamics for regression and classification problems using our simulated data. Results indicate that given a plume image sequence and associated laser parameters, we can not only predict which element the plasma was generated from with high confidence but also predict the set of thermophysical properties of the material. These results serve as proof-of-principle for plasma plume dynamics as strong predictors of fundamental material properties and motivate new experimental measurement techniques using laser ablation.

4:00pm PS+AIML-ThA-8 PSTD Business Meeting & Awards Ceremony,

Plasma Science and Technology Room Ballroom BC - Session PS-ThP

Plasma Science and Technology Poster Session

PS-ThP-2 Dependence of MQW Sidewall Damage on Ion Incident Angle: Insights from Molecular Dynamics Simulations, Eun Koo Kim, Hyun Woo Tak, Geun Young Yeom, Sungkyunkwan University (SKKU), Republic of Korea

Micro-LED (uLED) technology is emerging as a next-generation display solution due to its high brightness, energy efficiency, and scalability. In uLEDs, multiple quantum wells (MQWs) serve as the primary light-emitting layers, and preserving their optical performance is critically dependent on minimizing sidewall damage. As the lateral dimensions of uLEDs continue to shrink, the increasing proportion of sidewall area makes it critical to control the damage that is inevitably introduced during plasma etching processes. Such damage leads to increased non-radiative recombination at the sidewalls, which in turn significantly degrades the external quantum efficiency of the device. [1]

Although several approaches such as sidewall passivation using various materials and post-treatment techniques [2], as well as atomic layer etching (ALE), have been proposed to mitigate this issue [3], a comprehensive understanding of how the ion incident angle affects MQW sidewall damage remains lacking.

In this study, we employ molecular dynamics (MD) simulations to investigate the angle-dependent characteristics of ion-induced sidewall damage in MQW structures. Specifically, the analysis is categorized into (1) physical damage induced solely by ion bombardment and (2) damage resulting from ion bombardment after reactive radical adsorption—simulating typical RIE and ALE conditions. The simulations are designed to quantitatively evaluate the extent of structural damage in terms of penetration depth, surface roughness, sputtering yield, and dislocation formation, as functions of ion incident angle, ion kinetic energy, and ion dose.

The results of this study provide atomistic insights into the mechanisms of ion angle-dependent sidewall damage of MQWs and offer valuable guidance for optimizing plasma etching processes in advanced uLED fabrication.

References

- [1] Z. Liu et al., *Light Sci. Appl.* 14, 64 (2025).
- [2] Y. Zhang et al., *Micromachines* 14, 991 (2023).
- [3] W. Y. Ho et al., *Appl. Phys. Lett.* 123, 062102 (2023).

PS-ThP-4 Atomic Layer Etching of GaN Micro Light-Emitting Diodes with Different Sidewall Slope, Yun Jae Park¹, Geun Young Yeom, Hong Seong Gil, Jong Woo Hong, Sungkyunkwan University (SKKU), Republic of Korea

GaN-based micro light-emitting diodes (μ LEDs) are widely used in display technologies due to their high brightness and high endurance in harsh environment. However, during the reactive ion etching (RIE) process for device definition of GaN-based μ LEDs, damage to the activation layer or sidewalls can significantly degrade the device's external quantum efficiency (EQE). To mitigate this, various methods, such as optimizing the etch process or conducting post-etch processes (passivation insulator deposition, annealing, wet etching), have been studied to remove the damaged layer. However, more precise damage control techniques are needed as device dimensions shrink. [1]

In this study, we propose combination of wet etching with a plasma-based anisotropic atomic layer etching (ALE) process to remove sidewall damage induced by ICP-RIE. Tetra methyl ammonium hydroxide (TMAH) wet etching, commonly used to remove the damaged layer and to improve the etch profile in GaN-based μ LEDs, is dependent on the crystal orientation, causing changes in the sidewall angle during processing. We examined how well the optimized ALE process could be applied to remove remaining sidewall damage across various sidewall angles, which vary with TMAH treatment time. To analyze the effect of ALE, we observed changes in electrical and optical performance, confirming improvements in both EQE and I-V characteristics when sidewall damage was effectively eliminated. Furthermore, transmission electron microscopy (TEM) analysis revealed that the damaged lattice region near the sidewall had been removed,

supporting the physical recovery observed through electrical characterization. [2]

This study shows that sidewall angle, etching, and surface treatment all play an important role in enhancing μ LEDs performance. The results suggest that employing ALE to precisely control the sidewall can improve the efficiency of GaN-based devices.

References:

- [1] J.-H. Park, M. Pristovsek, W. Cai, H. Cheong, A. Tanaka, Y. Furusawa, D.-P. Han, T.-Y. Seong, and H. Amano, "Impact of sidewall conditions on internal quantum efficiency and light extraction efficiency of micro-LEDs," *Adv. Optical Mater.*, vol. 11, no. 10, p. 2203128, Mar. 2023
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PS-ThP-5 Anomalous Behavior of Plasma Potential in a Planar Helical Resonator Discharge, Un Jae Jung, Yeong Jae Jeong, Min Seok Kim, Chin Wook Chung, Hanyang University, Republic of Korea

Since a helical resonator plasma source does not require a matching network, it enables high-efficiency discharges and is considered a promising next-generation plasma source. An anomalous increase in plasma potential is observed in a planar helical resonator. As the applied power increases from 10 to 50 W, the plasma potential first increases and then shows a decreasing trend, similar to the E-to-H mode transition observed in inductively coupled plasmas (ICPs). Interestingly, as the power is further increased to approximately 400 W, the plasma potential increases again. The abnormal increase in plasma potential observed at high power disappears when a Faraday shield is inserted between the antenna and the plasma. This behavior is attributed to the significant amplification of the helical antenna voltage caused by resonance. These findings provide insight into the mechanism of plasma potential formation in planar helical resonator discharges and the role of antenna-plasma coupling.

PS-ThP-6 Low-damage Atomic Layer Etching process for GaN-based Light Emitting Diodes, Chan Ho Kim, Jong Woo Hong, Geun Young Yeom, Sungkyunkwan University (SKKU), Republic of Korea

Today, due to its wide direct bandgap and high efficiency, Gallium Nitride (GaN) based devices have gained significant attention in various applications such as light-emitting diodes (LEDs) and power semiconductors. As the critical dimension of LED devices becomes smaller, reactive ion etching (RIE) process is widely used to fabricate GaN based devices to achieve anisotropic profile. However, ion bombardment during RIE process causes surface damage, which deteriorates GaN based LED device performance. This problem becomes more significant as critical dimension of LED device becomes smaller due to the higher ratio of sidewall area to total area.

In this study, atomic layer etching (ALE) process is introduced after RIE process to remove damage caused by RIE process in GaN based structure, including multi-quantum well (MQW) layer composed of InGaIn and GaN. In TEM images, the MQW layer appeared indistinct after RIE process. Although wet etching followed by RIE improved layer visibility to some degree compared to RIE process, ALE process made the MQW layer more clear than wet etched MQW, indicating more appropriate damage removal. X-ray photoelectron spectroscopy (XPS) analysis exhibited that RIE induced damage changed the atomic ratios of N/Ga and Ga/In relative to reference data. However, after ALE process followed by RIE, the atomic ratios were returned similar to the reference data, although wet etch process also slightly restored atomic ratios. Furthermore, like XPS analysis data, Raman spectroscopy revealed that ALE process removed damage more efficiently compared to wet etch. Photoluminescence (PL) measurements at the same area showed that, as device size decreased, the damage caused by RIE is worse. However, PL intensity improvement was observed after ALE treatment and, as device size is smaller, the improvement in PL intensity is higher. Therefore, compared to wet etch process after RIE, ALE offers superior surface damage removal, especially showing its effectiveness in smaller devices.

PS-ThP-7 Comparison of SiN_x/SiO_x contact hole etching between CF₄ and low global warming gas, Jun Won Jeong, Geun Young Yeom, Jong Woo Hong, Sungkyunkwan University (SKKU), Republic of Korea

Demands for thinner, lighter, and higher-resolution panels in digital devices such as mobile phones, TVs, and laptops has led to the evolution of display technology such as LTPS (Low-Temperature Polycrystalline Silicon) technology. [1-2] LTPS thin film transistor (TFT) uses the excimer laser

annealing (ELA) for crystallizing amorphous silicon (a-Si) at lower temperatures, therefore, LTPS achieves significantly higher electron mobility than conventional a-Si. [3] In the device processing for next-generation LTPS TFT, optimizing the SiN_x/SiO_x stack contact hole dry etching process is critical. This requires high SiN_x/SiO_x stack etch rates, minimal sidewall damage, and anisotropic etch profiles, and, conventionally, CF₄ is generally used in the SiN_x/SiO_x stack contact hole etching. This study compares the conventional perfluorocarbon (PFC) CF₄ gas with low global warming potential gases in the dry etching of SiN_x/SiO_x stack contact holes.

By using low global warming potential gases instead of conventional CF₄ in the etching of SiN_x/SiO_x stack, little lower SiN_x/SiO_x etch rates compared to CF₄ were obtained, however, much similar etch selectivity between SiN_x and SiO₂ in addition to higher etch selectivity over photoresist could be observed. In addition, more anisotropic etch profiles of contact hole and the lack of microtrenching at the edge of contact hole could be obtained with low global warming gases. The etch mechanism could be confirmed by observing the plasma characteristics with OES and QMS, and by measuring the surface characteristics after etching with XPS.

Therefore, for the contact hole etch processing, it is believed to be possible to replace CF₄ having a high global warming potential with alternative low global warming gases with enhanced etch characteristics.

PS-ThP-8 Enhancing Etch Characteristics of MTJ using RF-Biased RIBE, Kyoung Chan Kim, Yun Jong Jang, Hong Seong Gil, Woo Chang Park, Dae Yeon Ha, Su Jeong Yang, Geun Yeong Yeom, Sungkyunkwan University, Korea

STT-MRAM is actively researched as a next-generation memory due to its non-volatility, fast operation, high stability, and ease of scaling, all of which are essential for high-performance computing and AI advancements. Materials such as CoFeB, Ru, MgO, etc. are used in the Magnetic Tunnel Junction (MTJ) layer for data storage in addition to CoPt and Colr to enhance magnetization stability. A common etching method for these MTJ stack layers is Ar⁺ Ion Beam Etching (IBE). However, the Ar⁺ IBE process leads to MTJ etch by-products redepositing on the pattern sidewalls. Tilting the substrate during Ar⁺ IBE is generally used to address this issue but does not fully resolve issues like shadow effects especially for recent high aspect ratio and small CD patterns. Previously, to address these issues, Reactive Ion Beam Etching (RIBE) has been investigated with reactive gases such as CO/NH₃ and Cl₂ to improve volatility of etch by-products. However, this can degrade the MTJ magnetization properties. RIBE process using H₂/NH₃ mixed gases has been also investigated to mitigate some of these issues.

This study aims to improve etching characteristics by using mainly physical etching with slight chemical assistance by RF-biasing. Ar gas is injected for physical etching while H₂/NH₃ mixed gas is injected on to the substrate for chemical effect. When RF power is applied to the substrate, the plasma of H₂/NH₃ mixed gas is discharged on the substrate and induces RF-Biased RIBE. SEM images were taken to analyze etch characteristics. TEM measurements were conducted to analyze the sidewall residues.

PS-ThP-9 Etch Characteristics of Ru-Pt Composite Using Halogen-Based Gases, Hyeon Joon Eoh, Geun Young Yeom, Sungkyunkwan University (SKKU), Republic of Korea

The lithography process is a key step in patterning, and it is one of the most challenging processes in the introduction of high-resolution semiconductor manufacturing. To overcome this challenge, advancements in photolithography technology have been progressing in the direction of utilizing shorter wavelength light sources. This has led to the development of Extreme Ultraviolet Lithography (EUVL), which is now used in a few nm processes. High-NA EUVL is a further refinement of this technology, enabling stable patterning even at sub-2 nm processes. As the photomask for EUVL, reflective mask containing patterned EUV absorbing layer is used. TaN-based EUV absorber is generally used as an EUV mask absorber but, for High-NA EUV systems, a new EUV mask absorbing layer is known to be required to reduce image distortion, which degrades pattern quality. Ru-Pt composite is a strong candidate to replace the TaN-based absorber, considering the above conditions. In this study, the etch characteristics of the Ru-Pt composite are examined using halogen-based plasmas. The Ru/Pt composition ratio was varied, and the corresponding etch characteristics were investigated.

When etching the Ru-Pt composite using fluorine-based gas in an ICP system, the etch rate increased with increasing Ru content in the Ru-Pt composite. In contrast, under chlorine-based gas chemistry, the etch rate increased with increasing Pt content in the Ru-Pt composite. In the case of pure Ar⁺ ion sputtering without halogen gases, the etch rate increased with

increasing Ru content in the composition. The effects of various process conditions on the etch characteristics of Ru-Pt composite required for EUV mask will be shown in the presentation.

PS-ThP-11 Isotropic Atomic Layer Etching of MoS₂ using Oxygen Plasma and Organic Solvent Vapor, Sunjae Jeong, Hyewon Han, Jieun Kang, Jimin Kim, Geunyoung Yeom, Sungkyunkwan University (SKKU), Republic of Korea
Precisely layer control of two-dimensional transition metal dichalcogenides (TMDs) is essential for the implementation of high performance electronic and optoelectronic devices. Atomic layer etching (ALE), which allows for precise layer control, can be performed using either thermal or plasma-based methods, enabling uniform etching. While conventional anisotropic etching has primarily been carried out through radical adsorption followed by ion-induced desorption, the increasing complexity of three-dimensional semiconductor device structures has led to a growing demand for isotropic etching techniques.

In this study, we utilize a method in which reactive radicals are generated through oxygen plasma and adsorbed onto the MoS₂ surface, followed by exposure to organic solvent vapor to facilitate the desorption of individual layers, thereby enabling precise layer control. Compared to conventional etching methods, this approach allows for damage-free processing while significantly improving the uniformity and precision of layer removal. Additionally, we compare the etching performance based on the chemical structure of the organic solvent vapor and the process temperature, emphasizing differences in reactivity and volatility during the etching process. These are important parameters in determining the efficiency and selectivity of the etching process. Our results confirm that the MoS₂ layers can be etched using a controlled manner, with approximately one monolayer removed per cycle, as verified through Raman spectroscopy and atomic force microscopy (AFM) analysis.

By achieving precise layer control of MoS₂, this study represents a significant advancement in the integration of TMD materials into next-generation electronic and optoelectronic devices. The findings contribute to the broader field of advanced materials research, paving the way for improved manufacturing techniques that meet the demands of future semiconductor technologies.

PS-ThP-12 Anticathode Effect on Multimodal Azimuthal Oscillations in Electron Beam Generated ExB Plasma, Nirbhav Chopra, Applied Materials, Varian Semiconductor Equipment; Yevgeny Raites, Princeton Plasma Physics Laboratory

Electron beam (e-beam) generated plasmas with applied crossed electric and magnetic (ExB) fields are promising for low-damage (gentle) material processing [1]. However, these plasmas can be subject to the formation of plasma non-uniformities propagating in the ExB direction. These rotating plasma structures (or 'spokes') enhance the transport of electrons and ions across the magnetic field, which can harm the gentle processing capability of plasma. In this work [2], we investigate the role of electrostatically active boundaries on the spoke formation by incorporating a variable bias conducting boundary (known as an anticathode) placed on the axially opposite side of the cathode. Our findings indicate suppression of azimuthal modes occurs when the anticathode is electron collecting. Furthermore, we show the highest frequency azimuthal mode is selectively suppressed by biasing the anticathode to an intermediate potential between the cathode and anode potentials. These findings suggest a link between the axial electron confinement in the e-beam generated plasma and azimuthally propagating plasma structure formation.

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PS-ThP-13 Plasmonic Plasma Process for Room Temperature Growth of Ultra-Thin Dielectric Films, Takeshi Kitajima, Machiko Miyake, Toshiaki Nakano, National Defense Academy, Japan

Catalytic surface reactions utilizing gold nanoparticle plasmons have been utilized in various applications in recent years.¹ We have applied hot electrons supplied from gold nanoparticles to plasma surface reactions to use them to form high-quality ultrathin films at room temperature.² We focused on the mixed effect of visible light for plasmon excitation and plasma VUV emission and discovered the effect of green light excitation that promotes radical nitriding. Due to the mercury probe measurement and TEM imaging, the film grown have superior dielectric feature and uniformity with less plasma induced damage in spite of nonuniform formation of gold nanoparticles. In the growth sequence, Au was vapor-deposited on a SiO₂/Si(100) substrate in an ultra-high vacuum chamber

with an average thickness of 0.4 nm by electron beam deposition to form Au nanoparticles (C) on the surface. A 30 mTorr N₂-inductively coupled plasma was generated in the attached chamber, and the sample was irradiated with N radicals (R) that passed through a 30 line/inch SUS304 single mesh with the configuration shown in Fig.1(a) for 5 minutes. A filter and a white LED controlled the wavelength of light (L), and VUV light from N₂ plasma was mixed. The reaction condition consisting of the above is RLC. Fig.1(b) shows the dielectric characteristics of the SiON film {leakage current and EOT (equivalent oxide film thickness) when 1 V is applied}. In green light suitable for Au plasmons, the hot electrons (~ 4 eV) generated by the deexcitation of plasmons enabled the bond conversion from Si-O to Si-N the ultra-thin SiON shows the same characteristics as the thermal oxide film. By mixing VUV, it is possible to increase the film thickness further and reduce leakage. Cross-sectional TEM image of SiON film after plasmonic process is shown in Fig.1(c). Beneath the Au particle SiON film with wide range of uniformity is confirmed and the single crystal lattice of Si substrate is clearly identified. Mixture of Au atoms into the dielectric film is examined with EDX spectrum shown in Fig.1(d). Au peak at 2.121 keV and 9.712 keV are less than the detection limit. From the above, it is considered that the reaction between the adsorbed N radicals and Si proceeded, and a good quality SiON film was formed by superimposing the photoelectron emission from the VUV light on the hot electron injection from the Au nanoparticles by green light irradiation.1 C. Clavero, Nat. Photonics 8, 95 (2014).2 T. Kitajima, M. Miyake, K. Honda, and T. Nakano, J. Appl. Phys. 127, 243302 (2020).

PS-ThP-14 Interaction of Sapphire (Single-Crystal Al₂O₃) and Ni-Based Alloy Surfaces with Halogen-Containing Plasmas and Gases, Takuya Ishihara, Hidenobu Tochigi, Azbil corporation, Japan; Hajun Kang, Osaka University, Japan, Republic of Korea; Kazuhiro Karahashi, Satoshi Hamaguchi, Osaka University, Japan

In semiconductor manufacturing processes such as dry etching or chemical vapor deposition, capacitance manometers are widely used as essential vacuum pressure sensors to monitor and control the pressures of process gases. These gauges must be corrosion-resistant against process gases such as halides and their radicals generated by the plasmas. The diaphragm material of the manometer is especially important because, if its surface is altered by such corrosive gases, the sensor would send imprecise output signals possibly with the zero-point drift or pressure sensitivity shift. The errors are caused by the changes in mechanical properties of the diaphragm arising from the formation of the modified surface layer. For this reason, Ni-based alloys or polycrystalline ceramics of aluminum oxide (Al₂O₃) are typically used as the diaphragm material of capacitance manometers. More recent capacitance manometers employ sapphire (single-crystal α -Al₂O₃) as their diaphragm material, which is of specific interest in this study[1]. Recent studies on the interactions of polycrystalline Al₂O₃ with fluorine-containing plasmas indicated the formation of aluminum fluoride layers on Al₂O₃ exposed to such plasmas[2,3,4,5,6]. We have reported the results of ion beam experiments to understand the surface modification mechanisms of Ni-based alloys and polycrystalline Al₂O₃ film by fluorine-containing plasmas[7]. In this study, similar ion beam experiments with sapphire substrates have been executed to compare the surfaces of single-crystal Al₂O₃ and polycrystalline Al₂O₃. In addition, Ni-based alloy samples were exposed to xenon difluoride (XeF₂) gases for 1,3,6, and 12 months, and their fluorinated surfaces were analyzed and compared with the sapphire surfaces under the same conditions reported previously[7].

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PS-ThP-15 3D Feature Profile Simulation with Realistic Plasma Chemistry for High Aspect Ratio Etching in the Memory Industry, Ju Won Kim, Seong Yun Park, Hae Sung You, Jae Hyung Park, Jeonbuk National University, Republic of Korea; Kook Hyun Yoon, Sung Sik Shin, Dong Hun Yu, KWTSolution, Republic of Korea; Yeon Ho Im, Jeonbuk National University, Republic of Korea

The memory industry has faced drastic challenges in high aspect ratio etching processes consisting of ON stacks (SiO₂/SiN) or SiO₂ to achieve higher cell densities or cell capacities. Recently, the problems encountered in HAR etching processes are mainly due to abnormal profiles such as necking, bowing, random distortion. Despite these difficulties, current process development is still largely based on trial and error due to the inherent complexities of plasma physics and chemistry and plasma-surface interactions. To address this issue, we have developed a 3D feature profile simulation platform called K-SPEED, which includes a zero-D reactor simulation, a multi-level set algorithm, a ballistic transport algorithm and a surface reaction model. The accuracy of this approach has been verified by the intensive comparative study of experimental evidence. In this work, we investigated the origin of abnormal profiles using realistic 3D feature profile simulation along with key process conditions. We believe that our process simulation platform will significantly help to optimise the HAR process for the next generation of memory devices.

PS-ThP-16 Plasma Anodization for the Production of AlF₃ Layers, Scott Walton, Naval Research Laboratory; Javier del Hoyo, NASA; Michael Johnson, Naval Research Laboratory; Luis Rodriguez de Marcos, NASA; Makenzie Meyer, John Murphy, Naval Research Laboratory; Manuel Quijada, NASA; Maria Sales, Virginia Wheeler, David Boris, Naval Research Laboratory

Efficient ultraviolet (UV) mirrors are essential components in space observatories for UV astronomy. Aluminum mirrors with fluoride-based protective layers are commonly the baseline UV coating technology; these mirrors have been proven to be stable, reliable, and have a long flight heritage. However, despite their acceptable optical performance, it is still insufficient for future large telescopes in which several reflections are required. Recently, a readily scalable, plasma-based passivation process was developed to produce a thin AlF₃ layer on the surface of aluminum. The passivation process uses an electron beam generated plasma produced in a fluorine-containing background (SF₆ or NF₃), to simultaneously remove the native oxide layer while promoting the formation of an AlF₃ layer with a tunable thickness. This process has the characteristics of established aluminum anodization approaches – either electrochemical or plasma – except here, fluorine replaces oxygen as the reactant. The process takes advantage of the ability of electron beam driven plasmas produced in electronegative gas backgrounds to generate substantial densities of negative ions, which can be delivered to the surface and utilized to grow the fluoride layer. While layer thickness scales with applied bias as expected, the growth rates are challenging to understand. In this presentation, we will discuss the process using operating parameter studies, plasma diagnostics, and materials characterization, with an eye on understanding the growth mechanisms and the potential for better process control. This work is supported by the Naval Research Laboratory base program and NASA grant no. NNNH20ZDA001N/20APRA200093.

PS-ThP-18 Experimental Investigation of the Interactions between Piezoelectric Crystals and Plasma Discharges, Jinyu Yang, Zhongyu Cheng, Sean Kerr, David Go, University of Notre Dame

Direct piezoelectric effect of non-centrosymmetric crystals, such as lithium niobate (LN) and lead zirconate titanate (PZT), provides opportunities to develop energy conversion plasma sources that remedy the need for high-voltage power supplies by directly transforming mechanical energy into plasma generation. To date, insight into the fundamental interactions and coupled physics between piezoelectric materials and plasma behaviors remains in its early stages. In this work, we utilized LN and PZT piezoelectric transformers (PTs) as model systems to investigate whether the level of the mechanically induced polarization in a piezoelectric crystal appreciably alters the behavior of a pulsed helium plasma jet impinging upon its surface, and whether these interactions manifest themselves in electrical characteristics. Preliminary optical and electrical characterization revealed that the morphology of the plasma jet plume and its contact at the plasma-crystal interface varied when the plasma jet generation was synchronized to different phases (i.e., different levels of polarization) of the input voltage to the PT. While no appreciable difference was observed in the plasma jet current, the current through the PT exhibited obvious suppression by the plasma jet, with the degree of suppression depending on the phase synchronization. Future studies will aim to achieve a more comprehensive

understanding of these phenomena using time-resolved imaging technology and to determine if the dominant plasma properties, such as electron density and electron temperature, are also sensitive to the changes in polarization. Experimental findings from this work will be compared with simulation results, assisting in the development of a multi-dimensional, pizeo-plasma coupled model.

PS-ThP-19 Tailoring of Pulse Voltage Waveform for Monoenergetic Ion Energy Distributions, *Seokhyeon Ha, Hyeonho Nahm, Minseok Kim, Heejae Yang, Chin-Wook Chung*, Hanyang University, Korea

Tailored voltage waveform on DC-pulsed bias has recently attracted interest as an effective means to control ion energy distribution functions (IEDFs). As the ion density increases, ion charging on the substrate increases, leading to a broadening of the IEDFs. For more advanced control of the IEDFs, a feedback system between the ion density and the applied voltage waveform is developed. To tailor the voltage waveform based on the measured ion density, a real-time ion density monitoring system is required. We employed a floating harmonic probe to measure the ion density in real time. Using the measured ion density, the slope of the voltage waveform is determined. This enables the IEDFs to remain narrow at various conditions

PS-ThP-21 Plasma-Assisted Uptake and Thermal Removal of Hydrogen in Liquid Lithium for Hydrogen Storage Applications, *Braden Moore*, University of Illinois at Urbana-Champaign; *Daniel O'Dea*, University of Illinois at Urbana-Champaign, UK; *Meenakshee Sharma*, University of Illinois at Urbana-Champaign, India; *Elliot Sherman, Zach Nordan, Loren Calleri, Riley Trendler, David Ruzic*, University of Illinois at Urbana-Champaign

Lithium hydride is a potential material for reversible hydrogen storage applications due to its high hydrogen content and energy density. Metal hydrides, in general, enable high mass density storage of hydrogen at low pressures and moderate temperatures, making them attractive for integration into future energy systems. While solid-state metal hydrides have been studied for this purpose, hydrogen uptake in liquid lithium for energy storage applications remains relatively underexplored. Hydrogen production through industrial-scale electrolysis or thermochemical splitting often involves high-temperature systems that could inherently maintain lithium in its molten state during hydrogenation. The University of Illinois at Urbana-Champaign (UIUC) has constructed the Actively Pumped Open-Surface Lithium LOop (APOLLO), which consists of a flowing lithium loop, a lithium free-surface, a hydrogen plasma source, and a distillation column for the thermal extraction of hydrogen. The flowing liquid lithium surface can be exposed to an Electron Cyclotron Resonance (ECR) hydrogen plasma source that has been characterized with an array of 16 Langmuir probes, a Retarding Field Energy Analyzer (RFEA), and actinometric spectroscopy. The hydrogenated liquid lithium then flows to an inductively heated Hydrogen Distillation Experiment (HyDE), which thermally treats the lithium at temperatures above 700°C to remove hydrogen. This presentation will focus on preliminary measurements of hydrogen uptake and removal at very low hydrogen concentrations. Future work will expand to higher hydrogen concentrations that are more applicable for an efficient energy storage system.

PS-ThP-22 Digital Twin Plasma Model for ICP Reactors: Integrated Multi-Physics and ML-Driven Optimization, *Muhammad Abdelghany¹*, Illinois Plasma Institute, University of Illinois at Urbana-Champaign; *Zachariah Ngan*, Department of Nuclear Engineering, University of California at Berkeley; *Dren Qerimi*, Illinois Plasma Institute, University of Illinois at Urbana-Champaign

Inductively Coupled Plasma (ICP) reactors are pivotal tools in modern semiconductor manufacturing, enabling high-precision etching and deposition processes essential for advanced device fabrication. Despite their widespread use, optimizing ICP reactor performance is challenging due to complex multi-physics interactions and the sensitivity of operating parameters, including RF power, frequency, gas composition, pressure, flow rates, and chamber geometry, as well as intricate plasma-surface interactions. We introduce a Digital Twin Plasma Model (DTPM) of an industry-grade ICP reactor that integrates multi-physics simulations, encompassing electromagnetic field computation, plasma kinetics, electron energy distribution, gas-phase chemistry, ion transport, and surface reaction kinetics, into a self-consistent framework. This high-fidelity model combines advanced physical models with machine learning-based predictive algorithms, providing a virtual replica of the reactor's plasma behavior.

The DTPM is implemented using a modular Python-based framework that defines the reactor geometry and plasma parameters and integrates an electromagnetic solver for computing inductive power coupling alongside a particle-in-cell (PIC) model for predicting ion density, electron temperature, and reactive species transport. Surface boundary conditions are incorporated to capture plasma-surface interactions on reactor walls and wafers. Ongoing validation uses a virtual probe to extract local electron temperature and plasma density, with a focus on capturing transient phenomena and non-uniform plasma distributions. To reflect the experimentally observed center-to-edge gradients, a 1D spatial resolution and a virtual Langmuir probe were incorporated into the kinetics solver, enabling direct comparison with experimental measurements. Preliminary results show qualitative agreement, indicating that the DTPM successfully reproduces these key features. In addition to physics-based simulations, the DTPM integrates machine learning (ML) techniques to enable surrogate modeling and real-time optimization. A data-driven surrogate model is trained on the simulation data, providing rapid predictions of plasma metrics as a function of control inputs. This ML-enhanced component accelerates parameter studies and supports on-the-fly optimization of operating conditions. By combining first-principles plasma modeling with ML-driven optimization, this digital twin approach paves the way for more efficient and adaptive control of next-generation industrial ICP systems in semiconductor production.

PS-ThP-23 Ion & Electron Energy Control with High Voltage Tailored Bias Waveforms in a CCP, *James Prager, Paul Melnik, Josh Perry, Chris Bowman, Timothy Ziemba, Kenneth E. Miller*, EHT Semi

The demand for solid-state non-volatile memory storage has increased the importance of plasma etching for producing high aspect ratio (HAR) features. To minimize defects in HAR features, precise control of the ion energy distribution function (IED) is essential. Additionally, controlling the electron energy distribution function (EED) is crucial to prevent positive charge buildup, which can distort etched features. EHT Semi has developed a high-voltage bipolar pulse generator that operates at 400 kHz. This system generates negative bias voltage waveforms that are flatter than those produced by standard sinusoidal radio-frequency generators, enhancing control over IEDs and process stability.

EHT conducted both experimental and computational studies to understand the interaction between bias waveforms and plasma properties. Using the bipolar pulser with a capacitively coupled RF plasma source, ion and electron energy distributions were measured with a retarding field energy analyzer (RFEA) at bias voltages up to 1.5 kV. Argon/oxygen plasmas were briefly investigated. The hybrid plasma equipment model (HPem) code was employed to create a computational analogue of the CCP chamber, further elucidating the system's capabilities.

PS-ThP-24 An RF Generator Driving an Inductively Coupled Plasma Source Without a Matching Network, *Timothy Ziemba, Chris Bowman, Paul Melnik, Josh Perry, Connor Liston, James Prager, Kenneth E. Miller*, EHT Semi

Inductively coupled plasma (ICP) sources are used throughout the semiconductor and thin film industries. ICPs are driven by a radio frequency (RF) generator that is impedance matched to the plasma. However, matching networks increase the cost, complexity, and thermal management requirements of ICPs, which all scale with power of the RF generator. Additionally, the breakdown is often unreliable and takes a significant amount of time.

EHT Semi has developed a new RF generator that eliminates the need for a matching network. This RF generator is being tested on ICPs across a range of experimental parameters (power, neutral pressure, and gases). EHT will present results on breakdown time and reliability compared to traditional RF generators with a matching network. The generator response to plasma impedance changes and constant and variable power will also be presented.

PS-ThP-25 Investigation of Ion Flux/Sidewall Interactions in High Aspect Ratio (HAR) Features, *Tanjina Akter, David S. Kanfer, Steven Shannon*, North Carolina State University

Ion interaction with vertical sidewalls in high aspect ratio etching plays a critical role in the etch profile of features in advanced memory devices. Feature distortions such as notching, bowing, and footing can occur due to deposition, sputtering, or charge accumulation brought about by ion interaction with these sidewalls. Charge accumulation is one of these interaction types that contribute to profile distortion. Simulations have

been conducted to spatially map this charge buildup, however, there is no diagnostic to provide experimental validation of this accumulation of charge. A novel diagnostic probe has been developed to measure the surface charge distribution inside the HAR features. The probe consists of an array of 10:1 aspect ratio vias (100 nm diameter) on PECVD Oxide with an aluminum ring encircling each via at varying heights. Voltage pickups from the aluminum rings enable the interpretation of a charge profile within the feature through differential measurement of voltage from an adjacent ring where the etched via is absent. This paper presents preliminary characterization of the ion particle flux and ion energy flux for an experimental CCP reactor that will be used to test this probe. A dual RF bias configuration with high (65 MHz) and low frequency (13.56 MHz) bias on each electrode has been employed to better control the ion energies. The control of ion flux distribution through the manipulation of the driving RF waveform can aid in mitigating charge-induced distortions and optimizing plasma processing for HAR structures. The IEDF was obtained by putting an RFEA on the lower-frequency electrode. Bimodal IEDFs were found for the pressure range of 1-100 mTorr, electron densities of $10^9 - 10^{11} \text{ cm}^{-3}$, and sheath potential of 50-1000 V using argon gas. The voltage at the top and bottom aluminum rings of a HAR via at the probe are calculated to be ~400 mV and ~100 mV respectively for the electron density of 10^{10} cm^{-3} and electron temperature of 4 eV, indicating that the design will have sufficient measurement resolution to measure these charge distributions.

This work is supported by the Department of Energy Office of Fusion Energy Sciences (DOE OFES Grant DE-SC0024545).

PS-ThP-26 Real Time Plasma Temperature Profiling Using Short Wave Infrared Imaging, Logan Holler, Drhuval Patel, Qerimi Dren, David Ruzic, University of Illinois at Urbana-Champaign; Michael Stowell, Lyten

Recent research has increasingly focused on the growth of graphene within atmospheric pressure plasmas. While it is well established that graphene formation is highly temperature-dependent, the distinction between the formation of graphene flakes versus nodules remains insufficiently characterized within plasmas. A key step forward centers on better mapping the temperatures across our different plasma mixtures. However, conventional diagnostic tools often fall short: most diagnostic systems only provide one-dimensional snapshots, and physical probes degrade rapidly under the high temperatures present in these environments.

To overcome these limitations, we propose the use of Short-Wave Infrared (SWIR) imaging as a nonintrusive method to obtain real-time, spatially-resolved temperature measurements across our plasma systems. SWIR imaging leverages blackbody radiation emissions to determine temperature by integrating spectral radiance over the detectable range of our camera. Provided that the camera's solid angle to the plasma remains fixed, changes in the integrated spectral intensity can be used to derive temperature ratios. By calibrating the system using a known temperature region within the plasma, we can correlate image intensity with absolute temperature for the same or similar plasmas by finding temperature ratios proportional to flux ratios. This allows for dynamic temperature mapping throughout the plasma, which is limited only to the refresh rate of the SWIR Camera.

Two methods are being developed to model and validate this approach. The first involves the insertion of a tungsten rod perpendicular to the flow of the plasma, which is incrementally raised through the plasma and allowed to reach thermal equilibrium at each position. This enables time-resolved images to determine temperature gradients and validate our current simulations. The second method involves varying gas mixtures to generate a calibration dataset, allowing the system to be adapted to different plasma environments. These experiments aim to correlate temperature regions with distinct graphene growth, such as flake versus nodule formation. Real-time, full-plasma monitoring also allows for characterizing how dynamic changes to the plasma occur, offering insight into the factors influencing graphene morphology.

PS-ThP-27 Monitoring Net CO₂ Dissociation Rates in the Effluent of Common Plasma Discharges with Optical Emission Spectroscopy, Andrew C Herschberg, Nathan Bartlett, Jameson Crouse, Jaime Robertson, Emily Greene, David N Ruzic, University of Illinois at Urbana-Champaign

Carbon dioxide is an important gas for many plasma discharges, among these include carbon capture and chemical conversion technologies. Such plasma-based systems offer increased sustainability by reducing net carbon footprints and limiting waste from industrial processes. During plasma excitation, much of the CO₂ present in the inlet flow will be reduced into CO

or other products. Therefore, the CO₂ dissociation fraction can be used as a metric for extent of reaction and to optimize process efficiency. Many methods can be employed for this purpose; in this work, an OES method of interest is compared against a standard QMS measurement. These metrologies are implemented into the exhaust gas from a flowing inductively coupled plasma containing CO₂ and N₂. The OES method employs a self-actinometry technique, comparing the line ratios from the CO Angstrom and N₂ second positive spectroscopic systems. This is implemented through a Gencoa OPTIX Remote Spectrometer for a more direct comparison to a differentially pumped SRS Residual Gas Analyzer. Overall both methods were comparable, measuring similar dissociation fractions under tested parameters, with a maximum dissociation of approximately 90%. Actinometric constants for the OES method were stable, deviating by as little as 2% across tested conditions. Implementation of the OES self-actinometric method will require calibration on system of interest, but showed to be more consistent with lower error than the QMS method.

PS-ThP-28 Measurement and Modelling of Sn-H₂ Vapor Diffusion Coefficients in the Transition Flow Regime, Jameson Crouse, Nathan Bartlett, Emily Greene, University of Illinois at Urbana-Champaign; Shiva Rajavalu, ASML; Andrew Herschberg, University of Illinois at Urbana-Champaign; Sergio Ferraris, Niels Braaksma, ASML; David Ruzic, University of Illinois at Urbana-Champaign

Extreme ultraviolet (EUV) lithography sources use tin in the process of generating 13.5nm wavelength light. Accurate modeling of neutral tin transport is important to understand how the tin coats different sections of the EUV source, which can reduce effectiveness. Modelling relies on knowledge of the diffusion coefficients of neutral tin vapor through molecular hydrogen. This work experimentally and numerically determined the diffusion coefficients of tin through molecular hydrogen at different tin temperatures and ambient pressures. Two experimental projects were used along with a CFD simulation and molecular dynamics simulation. For the most recent experiments, a known amount of tin is evaporated using an inductively heated crucible into a pipe with a known flow profile at a known ambient pressure. An OpenFOAM CFD model of the pipe is used to determine the flow profile within the pipe, along with the use of a diffusion model to predict tin transport. The pipe is inside of a large EUV source chamber prototype which can handle high hydrogen flows. Deposited tin is measured downstream of the pipe at varying distances with witness plates of different materials. Thickness measurements done with a profilometer are used to measure tin flux downstream of the Sn vapor source, which is then compared to the CFD model. Inertia and diffusion coefficients are adjusted in the model to match modelled fluxes to the experiment. A separate experiment is conducted utilizing mass loss measurements of a long crucible kept at a constant temperature and pressure over multiple hours, with tin evaporating out at a known rate. A variety of analytical and numerical coefficients were then compared to experimental fits of mass flux vs position to find the diffusion coefficient. Chapman-Enskog and Fick's law are the main analytical models utilized. A LAMMPS molecular dynamics model is also utilized to provide a wide array of results across the parameter space studied in this work, which is found to follow Chapman-Enskog theory well, even at lower pressures. The LAMMPS model uses mean squared displacement of Sn to calculate diffusion as the particles interact with H₂ through a Lennard-Jones potential. The results detail diffusion coefficients of tin in molecular hydrogen for varying temperatures, pressures, and hydrogen flow speeds with minimized error for each measurement and converging results between experiments."

PS-ThP-29 Simultaneous Deposition and Removal of Tin in a Hydrogen Plasma Environment, Nathan Bartlett, Jameson Crouse, Andrew Herschberg, Emily Greene, Jaime Robertson, Jack Granat, Lucia Suarez Heredero, Matias Habib, Karl Vu, David Ruzic, University of Illinois at Urbana-Champaign

Tin laser produced plasmas (LPPs) are used to generate 13.5 nm light in state-of-the-art extreme ultraviolet (EUV) lithography tools. Inside these tools, hydrogen gas is used as a buffer gas to decelerate ions from the LPP and is photoionized in the process creating a steady background hydrogen plasma. This plasma etches away tin as it accumulates on the wall of the EUV source forming the volatile compound stannane. Accurate etching rates of tin are needed to model tin accumulation inside of an EUV source. In this work, we present the results of a new experiment at the University of Illinois at Urbana-Champaign where tin vapor is simultaneously deposited and etched off of a substrate. In the experiment, a high temperature effusive source is used to deposit tin vapor onto a substrate while a hydrogen microwave plasma is used to generate hydrogen radicals

and remove tin from the surface. Etch rates are presented as well as the morphology of tin accumulated onto the substrate surface. The experiment is simulated using a transport and surface chemistry model ran in the OpenFOAM framework. Results from the experiment are compared with the model and used to validate the model.

PS-ThP-30 Plasma Cleaning Performance and GWP Reduction Using ClF_3 as an NF_3 Alternative in Semiconductor Manufacturing, Dong Ha Song, Sun Jae Jeong, Ga Hee Oh, Geun Young Yeom, Sungkyunkwan University (SKKU), Republic of Korea

The effective removal of process residues from the chamber following Si-based deposition is critical for ensuring process uniformity and maintaining tool performance in semiconductor manufacturing. Nitrogen trifluoride (NF_3) has been widely adopted for this purpose, but its high global warming potential (GWP) presents increasing environmental concerns. In response, chlorine trifluoride (ClF_3), a highly reactive gas with significantly lower GWP, has emerged as a promising alternative [1].

In this work, we evaluated the cleaning performance of ClF_3 compared to NF_3 in plasma-enhanced environments, focusing on both etch rate and spatial uniformity across the chamber. Silicon dioxide and silicon nitride-coated wafers were strategically placed within the chamber to quantify residue removal efficiency under both gases. To further improve gas utilization, we introduced nitrogen (N_2) as a diluent and analyzed the impact of its partial pressure on cleaning effectiveness.

The experimental results demonstrate that introducing N_2 allows for a reduction in overall gas consumption while maintaining comparable etch performance. Moreover, ClF_3 not only offers environmental benefits but also delivers efficient cleaning across various chamber regions, making it a viable low-GWP alternative for next-generation plasma cleaning processes [2].

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PS-ThP-31 EUV PR Patterned SiON Etching with Enhanced Selectivity and LER Control Using Ion Beam Technique, Yun Jong Jang, Hong Seong Gil, Kyoung Chan Kim, Woo Chang Park, Dae Yeon Ha, Geun Young Yeom, Sungkyunkwan University (SKKU), Republic of Korea

As extreme ultra-violet (EUV) lithography advances toward sub-10 nm patterning, the etch challenges associated with thin, low-resistance EUV photoresists (PR) become increasingly critical. This study presents a novel ion beam etching approach employing a grid pulsing technique to enhance the etch selectivity and line edge roughness (LER) control for EUV PR-masked SiON structures. In the proposed method, Ar/H_2 plasma is generated in an inductively coupled plasma as a source chamber, while a fluorocarbon gas mixture ($\text{CF}_4:\text{C}_4\text{F}_8$) is injected into the downstream process chamber. Pulsing the grid voltages modulates the ion energy temporally, alternating between high-energy etching and low-energy fluorocarbon deposition phases.

We observed that decreasing the pulse duty ratio from 100% to 50% significantly increased SiON-to-EUV PR etch selectivity (approaching ∞) and reduced LER from 9.6 nm (continuous mode) to 6.1 nm, closely matching the unetched reference (5.8 nm). XPS and FTIR analyses showed increased fluorocarbon presence at lower duty ratios, correlating with reduced PR damage and improved LER while maintaining etch profile quality. Ion energy distribution measurements confirmed the alternating high/low-energy ion bombardment mechanism as key to balancing selectivity and LER.

These results highlight grid pulsing as a powerful, tunable method for precision etching in EUV-based semiconductor processing, offering new opportunities for sub-10 nm node integration with minimized pattern distortion.

PS-ThP-32 Reduction of Plasma-Induced Damage via Ultra-Low Electron Temperature Plasma in Gate-All-Around FET Fabrication, Kim Hyungdong, Chung ChinWook, Nahm HyunHo, Ha Seokhyun, Hanyang University, Korea
 HfO_2 and TiN are representative materials used as gate oxides and metal gates in gate-all-around structures. However, damage caused by plasma during plasma processing has recently become a major concern due to its significant impact on device performance. In this paper, an ultra-low-

electron-temperature (ULET) plasma ($T_e < 1$ eV) is applied to improve the etch characteristics of HfO_2 and TiN. The ULET plasma is generated using an inductively coupled plasma with a DC-biased grid. Results show that the ULET plasma reduces surface roughness by 33% for HfO_2 and 38% for TiN compared to conventional plasma. The residual concentration of fluorine decreases by 14% for HfO_2 and 58% for TiN, indicating an improvement in surface impurity levels. The improvements are attributed to the lower ion energy in ULET plasma, which suppresses ion bombardment damage. Our findings demonstrate that mitigating plasma ion damage to HfO_2 and TiN effectively prevents performance degradation and expands the process window in semiconductor manufacturing, and enables the implementation of more diverse device structures.

PS-ThP-33 Micro-Masking Effects in TCP-ICP RIE with Fluorine-Based Plasmas: Influence of Alumina and Quartz Dielectric Screens, Giuseppe Libero Bufi, Pascual Muñoz, Daniel Pastor, Universitat Politècnica de València UPV, Spain

Surface quality is a key factor in microelectronics and photonics fabrication, where even slight increases in roughness can degrade optical or electrical performance. During plasma etching, micro-masking can lead to surface roughening and the formation of particle-like features, which are generally undesirable for high-precision patterning.

In this study, we investigated unexpected surface roughening during the etching of silicon dioxide (SiO_2) and silicon nitride (SiN) films in a transformer-coupled inductively coupled plasma (TCP-ICP) reactive ion etching (RIE) system using CF_4 and CF_4/O_2 gases. Despite testing different combinations of chamber pressure, source power, substrate power, and gas flow ratios, the etched surfaces consistently exhibited nanometer-scale roughness and micro-masked features with an average area greater than 200 nm^2 per feature. Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) revealed the presence of aluminum and fluorine on the etched surfaces, with an Al:F atomic ratio of approximately 1:3.6, indicating that the deposited material is likely aluminum fluoride (AlF_3). A plausible mechanism involves fluorination of the alumina protective screen located beneath the quartz window, followed by ion-assisted sputtering and redeposition onto the wafer surface, where the low volatility of AlF_3 under these conditions leads to persistent micro-masking. The dielectric screen serves to shield the quartz window from direct plasma exposure but can thus become a source of redeposition.

To assess the role of reactor hardware on surface quality, the 1 mm-thick alumina screen was replaced with a quartz screen of similar geometry. A direct comparison using the same CF_4/O_2 etch recipe on unpatterned 300 nm SiN-on-Si wafers showed a significant improvement: atomic force microscopy (AFM) measurements revealed a root-mean-square roughness R_q of 5.25 nm with the alumina screen, reduced to 0.49 nm with the quartz screen, representing a tenfold improvement. SEM inspections further confirmed the elimination of micro-masked surface features with the quartz dielectric screen.

This investigation identified the source of the micro-masking, enabling a screen material change that significantly improved the etch process. Beyond providing a practical mitigation strategy, this study underlines the importance of considering hardware-induced micro-masking in the development of roughness-sensitive microelectronic and photonic fabrication processes.

PS-ThP-34 Computational Study of a Radio-Frequency Positive Ion Source for Neutral Beam Injectors, Mohammad Sazzad Hossain, Amanda Lietz, Tom Regev, Arthur Mazzeo, Keanu Ammons, Florian Laggner, Miral Shah, Kirtan Davda, Steven Shannon, North Carolina State University; Evan Kallenberg, Brendan Crowley, Tim Scoville, General Atomics

The Large, Uniform Plasma for Ionizing Neutrals (LUPIN) is a radio-frequency (RF) inductively coupled plasma (ICP) chamber designed to demonstrate and optimize a positive ion source upgrade for the neutral beam injection system on the DIII-D tokamak. LUPIN is designed to operate at up to 20 kW of RF power at 2 MHz, coupling energy through a cylindrical quartz vessel (20 cm in length, 10 cm radius) to achieve target ion current densities of 1500 A/m^2 . This work presents fluid-kinetic modelling of a LUPIN H_2 plasma with parametric sweeps in RF power, pressure and frequency to study their impact on plasma chemistry, density, and ion flux at the grid. The effects of each operational parameter on plasma density, ion flux, and uniformity were investigated and the density was validated against experimental measurements. Increasing power shifts the primary ionization channel from molecular to atomic with diminishing flux increases due to skin-depth contraction and rarefaction, higher frequency localizes

heating and raises H_2^+ and H_3^+ delivery to the grid, while elevated pressure boosts ionization yet degrades ion transport due to increase in collisionality.

This work is supported by US DOE under DE-SC0024523 and DE-FC02-04ER54698.

PS-ThP-35 Amorphous Carbon Deposition: Insights from Plasma Dynamics, Feature Scale Modeling, and Density Functional Theory, *Purva Paranjape*, Purdue University, USA; **Kallol Bera**, Applied Materials Inc.; *Rupali Sahu, Nakul Nuwal, Sathya Ganta, Prashant Kulshreshtha*, Applied Materials, Inc.

With advances in nanoelectronics, 3D integration of semiconductor devices has become essential. Amorphous carbon gapfill is critical for enabling vertical stacking. Plasma Enhanced Chemical Vapor Deposition (PECVD) is widely used to deposit these sacrificial layers. Achieving conformal, void-free deposition is vital for structural integrity. Partial gapfill studies help identify process conditions that yield such profiles. Hydrocarbon-based PECVD at intermediate pressures shows non-monotonic deposition behavior: decreasing then increasing with pressure. At lower pressures, films exhibit overhangs and high bottom deposition. At higher pressures, deposition becomes conformal and void-free.

To understand these mechanisms, we use a multi-scale modeling approach combining 1D plasma simulations, 3D feature-scale modeling, and Density Functional Theory (DFT). The plasma model includes continuity equations, drift-diffusion for electrons, ion momentum conservation, energy balance, and Poisson's equation. A particle model with collisions is used to calculate ion energy and angular distribution using electric fields from the plasma model. Feature-scale simulations use Monte Carlo methods to model particle transport, surface reactions, and profile evolution. DFT provides adsorption energies of plasma-generated species on carbon surfaces, revealing the relative importance of surface processes.

Simulations show that flux of dissociation-generated radicals decreases, while polymerization-generated radicals increase with pressure. Ion flux remains stable, but ion energy drops and angular spread broadens. At lower pressures, deposition is ion-assisted via dissociated radicals. At higher pressures, chemisorption of polymerized radicals dominates. DFT shows stronger adsorption of dissociated radicals. Feature-scale modeling reproduces experimental profiles, validating our multi-scale model. Directional ion-assisted deposition causes overhangs at low pressure; chemisorption leads to conformal films at high pressure. A pinch-off occurs at intermediate pressures due to competing mechanisms. Our study highlights the role of plasma-surface interactions due to ion flux, energy, angular distribution, and neutral fluxes in controlling feature profiles. The validated model helps identify optimal conditions for desired feature profiles.

PS-ThP-36 Spatiotemporal Analysis of a Submerged Water Plasma Driven with Nanosecond Long Voltage Pulses, *Michael Johnson, David Boris, Lina Petrova*, Naval Research Laboratory, USA; **Mackenzie Meyer**¹, National Research Council; *Scott Walton*, Naval Research Laboratory, USA

Atmospheric pressure plasmas generate a distinct chemical and electrical environment ideal for treating water, making them attractive for applications in wound healing, chemical synthesis, nanomaterial fabrication, and water remediation. These plasmas can operate in a nonequilibrium regime when driven by short pulses of power, lasting tens to hundreds of nanoseconds, that energize electrons but are too short to significantly heat the surrounding gas. This study investigates the impact of pulse width on plasma-water interactions by applying 70–350 ns pulses to an argon plasma submerged in water. Plasma properties are analyzed using optical emission spectroscopy and electrical measurements. Results indicate that within the first 15 ns of the pulse, the plasma fully fills the gap between the electrodes. After this initial stage, the plasma expands to occupy the entire inter-electrode space for the remainder of the pulse, forming an arc-like plasma where current flow is regulated by the power supply. Essentially, pulse width determines how long the plasma remains in this high-current state. Optical emission spectroscopy revealed that argon dominates the emission immediately after plasma formation, but over time, hydrogen emission becomes more prominent as the plasma dissociates water molecules. This results in higher power consumption at longer pulse widths due to increased energy transfer to the water. Spatial emission profiles show uniform hydrogen emission across the reactor, whereas argon emission weakens near the positive electrode. Significant broadening of emission lines was observed during the pulse, with Stark broadening of

hydrogen lines used to estimate electron density. Measurements indicate that a substantial electron density persists for several microseconds after the pulse, likely due to residual voltage on the electrodes during power supply neutralization. At the longest tested pulse width (350 ns), the post-pulse current lasted nearly 10 μ s, highlighting not only the influence of pulse width on plasma dynamics but also the importance of other system parameters in determining plasma lifetime.

This work was partially supported by the U.S. Naval Research Laboratory Base Program.

PS-ThP-37 Reliable Monitoring of Plasma Chamber Cleaning via a Newly Developed Endpoint Evaluation Sensor, *Suyoung Jang, Dohyeon Kim, Kyongnam Kim*, Daejeon University, Republic of Korea

This study presents the development and application of a Cleaning Endpoint Evaluation Sensor (CEES) to enhance monitoring accuracy and uniformity control during NF_3 -based remote plasma cleaning in semiconductor equipment. Increasing chamber pressure increases fluorine radical density and improves etch reactivity, but cleaning non-uniformities persist, particularly in structurally constrained “dead volume” regions, such as the undersides of substrates, where reactive species cannot effectively reach. Conventional diagnostics, such as Optical Emission Spectroscopy (OES), lack the capability to verify cleaning completion in these challenging areas. In contrast, the CEES directly measures the removal of thin films that mimic actual process residues, providing real-time, spatially resolved endpoint evaluation. Experimental results demonstrate a strong correlation between CEES measurements and actual etch behavior, enabling the identification of regions where cleaning remains incomplete even when OES indicates completion. These findings highlight the potential of CEES as an effective tool for monitoring cleaning uniformity and determining the true endpoint of plasma-cleaning processes. Integration of CEES into advanced diagnostic platforms could significantly improve process reliability and efficiency in semiconductor manufacturing.

PS-ThP-38 Effect of H_2O Addition on Fluorocarbon Layer Formation on SiO_2 and Si_3N_4 Films in C_4F_8 Plasmas, *Haegeon Jung, Heeyeop Chae*, Sungkyunkwan University (SKKU), Republic of Korea

The effect of H_2O addition to C_4F_8/Ar plasmas on fluorocarbon (Cx_Fy) layer formation was investigated on SiO_2 and Si_3N_4 films and compared with O_2 addition. Tailoring of the fluorocarbon layer is a critical factor in achieving high selectivity and profile precision in high-aspect-ratio contact (HARC) etching. Etching was performed at 21 mTorr, 20°C, 30s, 130W source power, and no bias power. The fluorocarbon layer on SiO_2 was 26 nm in C_4F_8 -only plasmas. O_2 addition reduced the thickness to 6 nm, and H_2O addition reduced it to 21 nm. The fluorocarbon layer on Si_3N_4 was 8.9 nm in C_4F_8 -only plasmas. O_2 addition decreased the thickness to 3.9 nm, and H_2O addition increased it to 12.5 nm. Oxide surface promotes carbon scavenging to CO/CO_2 , and ion-assisted removal is strong, and the fluorocarbon layer on SiO_2 remains thin with bias [1,2]. Nitride surface lacks an efficient carbon-removal path to CO/CO_2 , and $NH_x/Si-F_x$ terminations increase CF_x retention, and ion bombardment cross-links and densifies the film, and the fluorocarbon layer on Si_3N_4 remains thick with bias [1,3]. SiO_2 surface provides polar sites that increase CF_x residence time and lower the nucleation barrier. CF_x nuclei survive more readily, and continuous-film formation is rapid. Interfacial $Si-F_x$ formation depletes F and weakens F-driven thinning, and the layer on SiO_2 becomes thick in C_4F_8 -only plasmas without bias [2,4,5]. Si_3N_4 surface shows slow initial nucleation, and F access is easy before continuity, and thickness increase is limited, and the layer on Si_3N_4 stays thin without bias [1,4]. H_2O addition increased the fluorocarbon layer thickness on Si_3N_4 and decreased it on SiO_2 . The increase on Si_3N_4 is attributed primarily to hydrofluorination-driven $NH_x/Si-F_x$ terminations that promote CF_x retention, and transient ammonium fluoro-silicate (AFS) formation may also occur under HF/NH_x -rich conditions, although it was not directly detected in this study [7,8]. O_2 addition inhibits fluorocarbon growth by oxidizing carbon species and scavenging CF_x radicals [6]. These results demonstrate substrate-dependent control of fluorocarbon growth by gas-phase H_2O , and sidewall passivation can be tuned precisely, and selectivity and profile precision improve in advanced etching applications.

PS-ThP-39 Data-Driven Sputtering Yield Prediction for DC Magnetron Sputtering, *Eunseo Lee, Hae June Lee*, Pusan National University, Republic of Korea

Plasma-based sputtering systems are widely used for physical vapor deposition of thin films. Among them, DC magnetron sputtering offers enhanced ionization efficiency through the magnetic confinement of secondary electrons near the target, leading to high deposition rates. A key

¹ JVST Highlighted Poster

process parameter in this system is the sputtering yield, which represents the number of atoms ejected per incident ion. The sputtering yield is known to vary depending on the ion energy, angle, and target surface condition, particularly as the target undergoes erosion during prolonged operation. In this work, we present a machine learning based framework for predicting sputtering yield variation during target erosion in a DC magnetron sputtering environment. To generate data for model training, we performed a particle-in-cell with Monte Carlo collisions (PIC-MCC) simulation for argon discharge. From the simulations, we extracted time-resolved ion energy and angular distributions (IEADs), as well as spatially varying plasma parameters, such as potential and density, near the target surface. The extracted plasma features were used as input data for a supervised neural network (NN) model. The sputtering yield corresponding to each condition was calculated using Yamamura's semi-empirical equation based on the incident energy and angle of argon ions. The trained NN model is intended to capture complex nonlinear relationships between plasma dynamics and surface response to enable fast estimation of yield variations without relying on time-consuming physical simulations. This approach is expected to serve as a practical tool for analyzing the impact of target erosion and optimizing process control in magnetron sputtering systems.

PS-ThP-40 Ultra-Low Temperature Dopant Activation Enabled by Plasma Enhanced Annealing, Zhihao Ma, University of Texas at Dallas; *Mahsa Shekarnoush, Yuanming Chen, Malcolm Bevan, Harvey Stiegler*, MicroSol Technologies Inc.; *Lawrence Overzet*, University of Texas at Dallas

The formation of highly doped, ultra-shallow junctions is critical for the continued scaling of advanced semiconductor devices. However, achieving high dopant activation while preserving lattice integrity remains a fundamental challenge.

Conventional annealing techniques such as furnace annealing and rapid thermal annealing (RTA) typically require processing temperatures above 800 °C to activate dopants and repair implantation damage. Although effective, these high-temperature treatments inevitably induce excessive dopant diffusion, junction broadening, and thermal degradation of fragile device materials, restricting their suitability for next-generation technologies.

Plasma Enhanced Annealing (PEA) has emerged as a compelling alternative, offering an ultra-low thermal budget through localized and controllable energy delivery from plasma ions. By tailoring ion energy, flux, dose, and species, PEA enables efficient dopant activation at substantially lower substrate temperatures (300–500 °C). This precise, near-surface energy transfer preserves shallow junction profiles, mitigates implantation-induced defects, and maintains device integrity while achieving high activation efficiency.

In this study, implanted silicon wafers were annealed using PEA, and their post-process properties were systematically evaluated using four-point probe measurements and Raman spectroscopy. Relative to conventional thermal annealing (TA) and RTA under identical conditions, PEA demonstrated noticeably superior electrical activation which is demonstrated by significantly reduced sheet resistance together with enhanced crystallinity. 500 °C PEA of shallow boron implants reached essentially the same sheet resistance as for RTA at 800 °C. Moreover, a comparative analysis of different inert ion species revealed distinct influences on dopant activation efficiency and lattice recovery.

These results establish PEA as a robust and versatile technique for forming highly activated, thermally stable, ultra-shallow junctions, positioning it as a strong candidate for integration into next-generation semiconductor device manufacturing.

PS-ThP-41 Tailored Waveforms for Ion Energy Control in ALE Applications, Sebastian Mohr, Hyungseon Song, Lucy Manukyan, Quantemol Ltd., UK

Atomic layer etching (ALE) is increasingly used in the manufacturing of semiconductor tools as they give more control over the resulting etching profiles than traditional etching techniques. While different approaches to ALE exist, many of them employ plasmas in one or more steps of the ALE process, be it to use the neutral radicals produced in the plasma to alter the surface or the ions to remove the altered top layer [1].

For such applications, independent control of ion flux and ion energy is highly desirable. Single frequency capacitively coupled discharges (CCPs) do not offer this, as the input power affects both flux and energy. Dual frequency discharges allow this to some extent, but it is limited due to, for example, increased ionization by secondary electrons at high powers of the low frequency. Furthermore, traditional CCPs usually produce bimodal ion

energy distribution functions which can cover several 10s to 100s of eV with sharp peaks at either end, so the ion energy cannot be easily limited to a small interval of energies, which is desirable especially for ALE applications, so that the ions remove the top layer of the surface but do not damage the underlying bulk [1].

An alternative approach to achieve this desired control are tailored waveforms. These can range from so-called asymmetric waveforms combining a fundamental frequency with even multiples [2] to non-sinusoidal waveforms typically consisting of sharp voltage peaks [1] followed by a relatively long interval of an almost constant voltage. While it has been demonstrated that these type of CCPs offer independent control of ion flux and energy and/or are able to limit the ion energy to narrow energy intervals, they have not yet been well studied in industrial applications.

This presentation will show continued efforts to simulate industrial applications of tailored waveform CCPs using the well-established 2D plasma simulation code HPEM [3]. In these discharges, the plasma is sustained via ICP coupling, while the tailored waveforms are applied to an rf-electrode staging the wafer. Former simulations have shown the intended effect in case of blank metal electrodes, i.e. almost monoenergetic IEDFs at the electrode. In the continued simulations, we investigate the effects of wafers on the produced IEDFs, for example via charging effects.

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Atomic Scale Processing Mini-Symposium Room 206 A W - Session AP+EM+PS+TF-FrM

Area Selective Processing and Patterning

Moderators: Steven M. George, University of Colorado at Boulder, Angelique Raley, TEL Technology Center, America, LLC

8:15am AP+EM+PS+TF-FrM-1 Exploring Intermolecular Interactions of an Acetylacetone Variant as Small Molecule Inhibitor for Area-Selective ALD, Eric Ha Kit Wong, Marc J. M. Merkx, Joost F. W. Maas, Eindhoven University of Technology, The Netherlands; Ilker Tezsevin, Eindhoven University of Technology, Netherlands; Wilhelmus M. M. Kessels, Eindhoven University of Technology, The Netherlands; Tania E. Sandoval, Universidad Tecnica Federico Santa Maria, Chile; Adriaan J. M. Mackus, Eindhoven University of Technology, The Netherlands

Self-assembled monolayer (SAMs) and small molecule inhibitors (SMIs) are two types of inhibitors for area-selective atomic layer deposition (AS-ALD). One critical requirement for inhibitors is to form densely packed adsorbate layers on the non-growth area. SMIs can be delivered in the vapor phase, which makes them compatible with existing industrial processes. However, the random sequential adsorption of SMIs tend to leave gaps in between the adsorbates (1). Intermolecular interactions could improve SMI packing and coverage, potentially leading to a higher selectivity.

π - π interaction is an important class of intermolecular interactions that has been employed in different fields (2). To exploit π - π interactions in SMIs, one can introduce aromatic backbones to the molecule. In this work, 3-phenyl-2,4-pentanedione (Hppa (3)) is used as the structural variant of acetylacetone (Hacac) for inhibiting ALD on Al_2O_3 as the non-growth area over the growth area of SiO_2 .

Using in-situ reflection-absorption infrared spectroscopy, we examined saturation behaviour, precursor blocking, and thermal desorption of Hppa. The results indicate that Hppa has similar adsorption behaviour as compared to Hacac, in terms of saturation dosages and bonding configurations. Still, various important differences are found: Firstly, the Hppa adsorbates exhibit a transition from a flat-lying configuration at low coverage, to a standing-up configuration at high coverage, suggested by the emergence of the $\text{sp}^2 \nu(\text{CH})$ peak. This indicates that the adsorbates are in orientations that could contribute to π - π interaction. Secondly, the results of blocking tests suggested that Hppa adsorbates are not displaced from the surface by bis(diethylamino)silane (BDEAS) precursor molecules, in contrast to the Hacac case in which ca. 5% of acac adsorbates are removed from the surface by BDEAS. Thirdly, Hppa adsorbates does not show thermal desorption at 150 °C for 10 hours. Instead, the increase in peak area suggest that the adsorbates might undergo rearrangement to other orientations. In summary, our results suggest that the Hppa can be an effective SMI for AS-ALD.

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8:30am AP+EM+PS+TF-FrM-2 the Influence of Intermolecular Interaction on the Packing of Small Molecule Inhibitors: A Simulation Study, Joost Maas, Marc Merkx, Eindhoven University of Technology, Netherlands; Tania Sandoval, Universidad Tecnica Federico Santa Maria, Chile; Adrie Mackus, Eindhoven University of Technology, Netherlands

In recent years, using small molecule inhibitors (SMIs) has become one of the main approaches to achieve area-selective atomic layer deposition (AS-ALD). The main challenge for area-selective deposition (ASD) using SMIs is to obtain a high packing of inhibitor molecules on the surface. Due to the vapor phase dosing, molecules arrive one-by-one at random locations, which leaves gaps in between the inhibitors where potentially a precursor can adsorb, causing loss of selectivity.[1]

In this work we explore the use of attractive intermolecular interactions for achieving higher packing of SMIs via simulations. When employing small molecules, it is expected that van der Waals interactions are too weak to contribute to packing. Consequently, one of the main questions of this work is how strong the interaction has to be to facilitate ordering of molecules on the surface. In addition, the synergy of intermolecular interaction and diffusion of SMIs over the surface is investigated.

The simulation method used for studying the packing of molecules with intermolecular interaction and diffusion is an augmented random sequential adsorption (RSA) model.[1] During every loop of the RSA model, either adsorption or diffusion takes place, based on the adsorption and diffusion rates. The intermolecular interaction energy is implemented in the form of a Metropolis-Hastings algorithm.[2] In the initial work, the molecule is a simple disk on a hexagonal grid with periodic boundary conditions, considering nearest neighboring blocking.

It was found that the coverage of the SMI improves significantly for low diffusion barriers and attractive intermolecular interaction. For interactions energies stronger than approximately -0.15 eV, the formation of domains of higher density on the surface is observed. The results suggest that coverage can be improved significantly by selecting SMIs with sufficient interaction energy and a low diffusion barrier.

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8:45am AP+EM+PS+TF-FrM-3 Tuning Surface Reactivity by Small Molecule Modifiers in Area-Selective ALD: Small Molecule Inhibitors (SMI) vs. Small Molecule Promoters (SMP), Andrew Teplyakov, University of Delaware

In area-selective deposition, selectivity of surfaces could be manipulated to either suppress or promote surface reactivity with respect to the target reactants. Using model ALD processes with TiO_2 (TDMAT/water) or Al_2O_3 (TMA/water), the deposition onto semiconductor surfaces modified with small fluorine-containing molecules is analyzed by spectroscopic and microscopic techniques, including depth profiling with ToF-SIMS, supplemented by computational DFT modeling. The fluorinated functional groups are designed for easy spectroscopic characterization to analyze the potential AS-ALD schemes on silicon, as well as on oxide materials, including TiO_2 , MgO , and Al_2O_3 . The initial deposition steps are analyzed by comparing the behavior of modified surfaces with that of pristine substrates, and the distribution of the fluorine and fluorine-containing fragments within the ALD-deposited layers is followed by ToF-SIMS depth profiling once these F-containing functionalities are buried under the overgrown layers. This approach allows for identification of the deposition processes for both small molecule inhibitors (SMIs) and small molecule promoters (SMPs).

9:00am AP+EM+PS+TF-FrM-4 Topographically Selective Atomic Layer Etching of HfO_2 and ZrO_2 Using NbF_5 and TiCl_4 , Boyun Choi, Getasew Zewdie, Hyeyoung Shin, Nari Jeon, Chungnam National University, Republic of Korea

As transistor dimensions continue to shrink, conventional SiO_2 gate dielectrics no longer provide adequate capacitance or leakage control, leading to the widespread adoption of high-k oxides such as HfO_2 and ZrO_2 . Yet, achieving atomic-level patterning of these oxides remains a key challenge for advanced device integration. In this study, we investigate their thermal atomic layer etching (ALE) behaviors using NbF_5 and TiCl_4 as representative halide reactants. A pronounced contrast emerges: HfO_2 undergoes smooth, self-limiting etching cycles, whereas ZrO_2 exhibits surface roughening and substantial chlorine incorporation. Density functional theory calculations clarify this difference by showing that TiCl_4 reacts more aggressively with ZrO_2 surface species, destabilizing the surface and degrading etch quality. Extending ALE to nanohole structures with a diameter of 150 nm and a depth of 2000 nm, we further observe topographically selective removal of HfO_2 . This selectivity is linked to microstructural factors such as crystallinity, indicating that not only chemical reactivity but also structural variations critically influence ALE outcomes. Taken together, these results establish a mechanistic framework for understanding how reactant chemistry and microstructure jointly govern the etching of high-k oxides. Such insights provide practical guidelines for enabling selective integration of HfO_2 and ZrO_2 in next-generation transistors and interconnect architectures, where atomic-scale precision and material specificity are indispensable.

9:15am AP+EM+PS+TF-FrM-5 Area Selective ALD for Future Engineering Challenges, Stacey Bent, Stanford University INVITED

The continued downscaling of electronic device dimensions requires the development of new, precise patterning methods that are compatible with high-volume manufacturing. Atomic level processing, and in particular area selective atomic layer deposition (AS-ALD), continues to gain attention as an important method to achieve nanoscale features at the sub-10 nm length scale. It is well known that tuning the surface chemistry of the substrate can be used to either inhibit or enhance ALD nucleation, leading to selective deposition. A key strategy for AS-ALD has been the use of

inhibitors which can alter the native surface reactivity to block nucleation in thermal as well as plasma-assisted ALD. This inhibition approach enables good selectivity in AS-ALD of thin films on a variety of substrate materials, including dielectrics and metals, and I will present several inhibitor-based AS-ALD systems. Importantly, the ALD precursor also plays a key role in influencing selectivity. Results show that precursor size can have a significant influence on the ability of inhibitors to prevent ALD nucleation. However, precursor size alone is not the defining metric, and I will share examples that highlight the influence of other precursor effects, such as precursor-inhibitor reactivity and miscibility. Ultimately, developing molecular design rules for both inhibitors and ALD precursors will be critical for applying AS-ALD more widely to future challenges in microelectronics fabrication.

9:45am AP+EM+PS+TF-FrM-7 Controlling ASD of a Multi-Color System: PEDOT ASD between SiN, Si-H, and SiO₂ by Pre-Treatment Adjustment, Jeremy Thelven, Nicholas Carroll, Gregory Parsons, North Carolina State University

Complex 3D device architectures are proposed as the solution to make devices more energy efficient.¹ These architectures require many lithographic steps where the high costs of EUV lithography limits device throughput. As such, there is a need for process augmentation to reduce the EUV burdening. A potential solution is area-selective deposition (ASD), where film deposition occurs on a “growth” surface while it is inhibited on an adjacent “non-growth” surface allowing for bottom-up processing.

While ASD conveys the notion of selective deposition between two surfaces, however, in fabrication more might be exposed. Therefore, it is crucial to look at multiple surfaces, a “multi-color system.” The goal being to have the versatility of depositing the desired material only on the desired location(s). It is then important to know processes that activate or deactivate specific surfaces in a multi-color system.

Poly(3,4-ethylenedioxythiophene) (PEDOT) was deposited by oxidative chemical vapor deposition (oCVD) using 3,4-ethylenedioxythiophene (EDOT) monomer and antimony pentachloride (SbCl₅) as reactants to analyze how various pre-treatment strategies can tune the ASD between Si-H, SiN, and SiO₂ surfaces. As a control, single-material coupons were treated with a diluted hydrofluoric acid (DHF) wet etch prior to PEDOT oCVD. Results showed ~30nm of ASD on SiN and SiO₂ vs. Si-H. Three different pre-treatment strategies were evaluated: 1 cycle of molybdenum hexafluoride (MoF₆)/N₂, 7 cycles of N,Ndimethylamino-trimethylsilane (DMATMS)/N₂, and 7 cycles of DMATMS/N₂ followed by a water soak. Ellipsometer, water contact angle, XPS, and SEM results show that MoF₆ served to simultaneously activate and deactivate the Si-H and SiO₂, respectively, allowing for PEDOT ASD on SiN and Si-H vs. SiO₂. DMATMS exposure deactivated only the SiO₂ showing PEDOT ASD on SiN vs. SiO₂ and Si-H. Including a water soak after the DMATMS activated the Si-H to PEDOT deposition resulting in a deposition configuration of Si-H and SiN vs. SiO₂.

Overall, the concept of tunable selectivity for a three-color system is demonstrated by these results. These pre-treatment strategies providing a better understanding into controlling selectivity.

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10:00am AP+EM+PS+TF-FrM-8 Kinetics Model for Selective Thermal Etching of Si_{1-x}Ge_x in F₂/Ar, Yi Chen, Daniel Cho, University of California, Los Angeles; John Hoang, Nicholas Altieri, Ji Zhu, Samantha Tan, Lam Research Corporation; Jane Chang, University of California, Los Angeles

The selective etching of Si_{1-x}Ge_x over Si enables the fabrication of the gate-all-around field-effect transistors. Thermal etching of Si/Si_{1-x}Ge_x at near room temperature features high selectivity, exhibiting a non-linear relationship between etch rate and Ge% (Fig. 1(a)). There are no reported reaction mechanisms explaining this unique Ge%-dependent phenomenon.

In this work, thin films of Si_{1-x}Ge_x of varying Ge content (Ge% = 0 to 1) were etched thermally by molecular F₂ gas at near room temperature under different F₂ partial pressures (0.5 to 10 mTorr) in Ar. The etch rates were quantified by ellipsometry measurement and the relationship between etch rate and Ge% resembled those shown in Fig. 1(a). Reported Si_{1-x}Ge_x etch selectivity ranges from 100 to 1000 and the unpublished maximum etch selectivity is from 200 to 250. The unpublished experimental data is being reviewed for public release and will be presented at the conference. A kinetics model was established in this work to elucidate the reaction pathways in thermal etching of Si_{1-x}Ge_x by F₂, considering reactions

between atomic fluorine and various surface species and the interplay between reaction products involving Si and Ge. The model result (Fig. 1(b)) yielded the unique volcano-shaped relationship between etch rate and Ge%, validating the reactions considered in the model captured the main kinetics during F₂ etching of Si_{1-x}Ge_x.

10:30am AP+EM+PS+TF-FrM-10 Area-Selective Deposition by Surface Engineering for Applications in Nanoelectronics: Enablement of 2d and 3d Device Scaling and Self-Alignment, Silvia Armini, IMEC Belgium INVITED

At advanced nodes targeting 10 nm feature size and below, lithography starts to dominate costs (EUV, multiple mask passes per layer, pattern placement error,...). Complementary techniques and materials are needed to continue 2D scaling and extend the Moore's law. On the other hand, 2D scaling is reaching its limitations driving the transition to 3D and vertical integration schemes (such as 3DNAND, 3DDRAM, CFET...), which result in higher devices density per unit area and lower production cost. Area-selective atomic layer deposition (AS-ALD) is rapidly gaining interest because of its potential application in self-aligned fabrication schemes for next-generation nanoelectronics. In addition, ASD allows coping with high aspect ratio and complex 3D architectures. The strong sensitivity of ALD to surface chemistry and its self-limiting nature are particularly appealing for ASD.

In this talk I will illustrate a variety of ASD processes and applications spanning from nano-interconnects, logic and memories to patterning.

11:00am AP+EM+PS+TF-FrM-12 MO-Mo? Oh No! The Problem of Carbon in Metalorganic Molybdenum Deposition, Kyle Blakeney, David Mandia, Matthew Griffiths, Jeong-Seok Na, Raihan Tarafdar, Jeremie Dalton, Lam Research Corporation

Molybdenum (Mo) halides and oxyhalides comprise the sole class of precursors that can deposit Mo metal films by ALD/CVD with sufficient purity for applications in advanced microelectronic devices. Unfortunately, solid, low vapor pressure Mo chloride precursors have challenges in flux stability and low vapor pressure. Metalorganic (MO) precursors are commonly used to address some of these challenges and are useful alternatives to halides for many non-metal films such as SiO₂, SiN, TiN, Al₂O₃, etc. Despite much effort, MO-precursors have not met the performance of chloride precursors for depositing pure Mo.

This presentation will summarize key findings of MO-Mo process development by the Lam ALD/CVD Metals concept and feasibility (C&F) group. Included will be typical precursor tests using coupon process modules, 300mm C&F chambers, fundamental mechanistic investigations of Mo surface reactivity, and novel deposition pathways such as conversion-reduction (Figure 1) and alloy formation (Figure 2).

11:15am AP+EM+PS+TF-FrM-13 The Effects of Process Chemistry on Blocking Chemisorption in ALD: Thin Film Precursor, Co-Reactant and Co-Adsorbate, Jay Swarup, James Jensen, Jeffrey Gao, James Engstrom, Cornell University

Achieving area selective deposition requires preventing growth on the non-growth surface (NGS), which often involves the use of molecules to block growth on those surfaces. Careful choice of the ALD process chemistry, thin film precursor and co-reactant, as well as the blocking molecule and how it is administered, is important. We report here a systematic examination of the effects of the precursor, co-reactant and co-adsorbate/blocking molecule on preventing growth of Al₂O₃ on SiO₂. We also consider the effects of temperature, and the dosing sequence employed for the blocking species. Concerning the precursor we compare trimethylaluminum (TMA) to a non-pyrophoric precursor containing only Al-N bonds and no Al-C bonds, i.e., BDMADA-Al [1]. For co-reactants we compare H₂O to *t*-BuOH. Finally, we consider two blocking species: octadecyl trichlorosilane (ODTS), and dimethylamine trimethylsilane (DMATMS). In this study we employ a quartz-crystal microbalance to monitor ALD *in situ* and in real-time, and the deposited thin films have been characterized *ex situ* using X-ray photoelectron spectroscopy, and a variety of techniques. Concerning the “pristine” processes, i.e., ALD in the absence of a blocking molecule, the properties of the films (density, C incorporation, stoichiometry, growth rates) are comparable using either BDMADA-Al or TMA as the precursor under similar reaction conditions. These species also react similarly with H₂O and *t*-BuOH as the co-reactant, where steady growth with the latter is only observed at sufficiently high temperatures. Concerning blocking growth, we have observed a number of identifiable trends. First, employing the same ALD process chemistry, ODTS produces better blocking in comparison to DMATMS in cases involving a single dose of the blocking molecule. When comparing TMA and BDMADA-Al, we observe that for

both blocking molecules that the latter is more efficiently blocked. These two observations demonstrate the importance of molecular size as the larger BDMADA-AI is more efficiently blocked, and the larger ODTs is better for preventing growth. Temperature has a definitive effect on the efficiency of preventing growth where we find that higher temperatures lead to more effective blocking of growth. The dosing sequence employed for the blocking species also plays an important role. Repetitive dosing of DMATMS in an "ABC" process provides superior blocking with respect to a single pre-exposure, and these results exceed those produced by ODTs.

[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).

Plasma Science and Technology

Room 201 ABCD W - Session PS1-FrM

Plasma Processes for Coatings and Thin Films

Moderators: Erwin Kessels, Eindhoven University of Technology, the Netherlands, Scott Walton, Naval Research Laboratory

8:15am **PS1-FrM-1 First-Principles Study on Film Stress Mechanisms of Amorphous Carbon: The Role of Bond Hybridization**, *Yusuke Ando*, Nagoya University, Japan; *Hu Li, Jianping Zhao*, Tokyo Electron America, Inc.; *Masaaki Matsukuma*, Tokyo Electron Technology Solutions Ltd., Japan; *Kenji Ishikawa*, Nagoya University, Japan; *Peter Ventzek*, Tokyo Electron America, Inc.

Amorphous carbon (a-C) is a highly versatile material with tunable properties, including hardness, electrical conductivity and optical transparency, which can be tailored through control over its fraction of hybridized bonds and its content of hydrogen. Among a-C materials, hydrogenated amorphous carbon (a-C:H) has been widely utilized as an etching hard mask in semiconductor fabrication processes due to its superior resistance to fluorinated gas plasma and its facile removal via oxygen plasma treatment.

With the continuous advancement of semiconductor fabrication technology, particularly in 3D flash memory devices, increasing number of stacking layers necessitates the development of high-aspect ratio etching techniques. To meet this requirements, a-C hard masks must exhibit enhanced etch resistance to withstand prolonged plasma exposure while maintaining controlled residual stress to prevent delamination and wafer bending. a-C films are typically deposited via plasma enhanced chemical vapor deposition (PECVD) with hydrocarbon-based plasma, and experimental observation shows that bias voltages promoted an increased sp³-C fraction and higher film density, thereby improving etch resistance. However, this increase in density is also accompanied by elevated residual stress, presenting a critical trade-off between etch resistance and mechanical stability. A fundamental understanding of stress generation and relaxation mechanisms is essential for optimizing a-C hard masks for advanced semiconductor applications.

While experimental investigations have provided valuable insights into stress behavior, the underlying structural factors governing stress generation remain insufficiently understood from a theoretical perspective.

In this study, as a first step, we have systematically analyzed influence of hybridized bonding configurations on residual stress of carbon films. By employing first-principles calculation, we modeled and evaluated various defective diamond-like carbon structures with identical densities, allowing us to isolate and compare the effects of geometrical properties other than density on stress generations. Our results indicate that, to varying degrees, a lower sp³-C fraction and shorter mean bond length contribute to increased compressive stress. These conclusions are tested on hydrogenated systems as well. Based on these findings, we propose a mechanism of stress-relief that can guide process optimization in fabricating high-performance a-C hard masks.

8:30am **PS1-FrM-2 Atmospheric Microplasma-Driven CVD for Highly Crystalline Carbon Nanotube Synthesis**, *Guohai Chen, Takashi Tsuji, Shunsuke Sakurai, Don Futaba, Kenji Hata*, National Institute of Advanced Industrial Science and Technology (AIST), Japan

Microplasma-assisted chemical vapor deposition (CVD) provides a powerful platform for advancing carbon nanotube (CNT) synthesis by enabling localized, high-energy reactions that promote controlled nucleation and growth, essential for harnessing CNTs' extraordinary properties across diverse applications [1-5]. We present a multi-step atmospheric microplasma CVD system that incorporates abrupt interaction steps to

precisely initiate and terminate nanoparticle (NP) aggregation through the coordination of microplasma and carbon reactant gas flow, thereby achieving the synthesis of highly crystalline CNTs [6-9].

Using this platform, we systematically investigated the roles of hydrogen and catalyst precursors under microplasma conditions. Hydrogen was found to play a critical role in moderating plasma chemistry, as revealed by a simple reaction pathway model: without hydrogen, excessive electron-induced decomposition suppressed catalyst NP formation and caused catalyst deactivation; with hydrogen, energy transfer shifted toward thermal pathways, enabling controlled NP formation and subsequent CNT nucleation. Optical emission spectroscopy validated these mechanistic insights [8]. We also studied the effect of catalyst precursor ligands on NP growth kinetics and CNT quality. Iron pentacarbonyl (Fe(CO)₅) produced smaller, more uniform NPs and resulted in higher-purity CNTs with greater yield compared to ferrocene (Fe(C₅H₅)₂), despite similar CNT structural features [9]. These findings highlight the significant influence of both hydrogen and precursor chemistry on catalyst behavior under microplasma conditions.

In addition to synthesis, we briefly introduce practical CNT applications, including a neural probe based on a mm-tall, high aspect ratio (60:1) CNT post array [10], and a through-silicon-via interposer integrating CNT-Cu composites, offering copper-level electrical conductivity and silicon-level thermal expansion [11].

Our study demonstrates the potential of atmospheric microplasma for precise, tunable CNT synthesis, offering new pathways for nanomaterial fabrication through plasma process engineering.

Keywords: Carbon nanotube, microplasma, crystallinity, precursor, optical emission spectroscopy

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Please see the Supplementary Document for the figure and reference list.

8:45am **PS1-FrM-3 AVS John Thornton Award Talk: Creating a Dream Team: Thin Films, Plasma Chemistry, Holistic Approaches, and Non-Traditional Pathways**, *Ellen R. Fisher*, University of New Mexico **INVITED**

Today, plasma processing is a well-known and powerful technique to modify the surface of materials, and create new materials, especially thin films. John A. Thornton was a pioneer in developing plasma processing of thin films. He was also a dedicated educator, having mentored numerous students. In this presentation, we present a holistic approach to plasma processing of thin films, linking the gas phase, the gas-surface interface, and relevant materials characterization. Often, the ultimate goal of these plasma-modification studies is to explore various pathways to tune and tailor the surface of a material, while maintaining bulk properties and material integrity for a desired application. Specific systems discussed will include semiconductor materials, membrane coatings, and metal oxides. Similarly, the development of the next generation of inventors and discoverers requires alternative approaches and new tools. To that end, the use of the science of team science (SciTS) tools and approaches provides alternative pathways to tune and tailor the environment necessary for creating effective teams. This can be realized by being the Archintor™ of a team's networks. Social network analysis (SNA) allows teams to discover how team members connect, including through learning, collaboration, and leadership networks. Fundamental SNA concepts and Archintor™ examples will be presented from real science and engineering teams, providing unique insight into the development and deployment of productive teams. Collectively, these studies exemplify the comprehensive approach to solving challenges in the plasma community, a tribute to the legacy of John A. Thornton.

Plasma Science and Technology

Room 201 ABCD W - Session PS2-FrM

Plasma in EUV Scanner Technology

Moderators: Erwin Kessels, Eindhoven University of Technology, the Netherlands, Scott Walton, Naval Research Laboratory

9:15am **PS2-FrM-5 Measurement of Cold Spit Tin Particle Trajectories in a Hydrogen Plasma**, *Jaime Robertson*, University of Illinois Urbana-Champaign; *Raoul de Rooij, Andrei Yakunin, Victoria Voronina*, ASML, Netherlands; *David Ruzic*, University of Illinois at Urbana-Champaign

The extended exposure of tin (Sn) particles to hydrogen radicals has demonstrated the ejection of sub-micron sized particles from a surface

covered in micron sized Sn droplets. This work experimentally investigates the three mechanisms, spitting, etching, and particle lift off, behind the mass transport of Sn, focusing on measuring the size, velocity, and directionality of the particles and the frequency of particle migration. Using a quartz crystal microbalance (QCM), measurements of the mass flux from a surface covered in micron sized droplets of Sn were performed with plasma radical densities similar to and greater than in the scanner. While etching rates of Sn by hydrogen were measured similar to other reports, significant, acute decreases in the mass were also observed throughout the duration of testing associated with either spitting or particle lift off. Further imagery, on a SEM, of the surface before and after exposure to hydrogen plasma confirmed a change in surface morphology. Larger Sn droplets appeared to have cratering along the surface, believed to be the result of cold spitting Sn. This is due to buildup of hydrogen beneath the surface forming a pressure gradient within the Sn particles leading to surface fracturing and particle ejection. An additional test was designed with a silicon wafer placed above the surface of the Sn droplets that captured cold spit particles. An SEM was then used to measure the size and position, allowing for the interpretation of mass and directionality of Sn leaving the surface. Velocity of spit particles is being determined by measuring the deflection of the cold spit particles through an electric field, having a known charge buildup on the surface. Based on the distance traveled before reaching the chamber walls, the incident velocity of particles is calculated. Measurement of liquid spit particles is underway using aerogel, with diagnostic techniques like ballistics work. The liquid particles are captured in the aerogel before using a uCT to generate a 3D rendering of the surface. Based on the cratering characteristics such as depth and width found at the surface of the aerogel, velocity of each particle is calculated. Additionally, this work is being performed on various surface materials to determine how the preferential recombination of hydrogen radicals with various surface materials impacts the rate of degradation of the Sn. Initial tests revealed a reduction in mass loss rates for gold when compared to aluminum. This is likely due to the greater recombination coefficient of gold when compared to aluminum.

9:30am PS2-FrM-6 Benefits and Challenges of Plasma in EUV Lithography Scanners, *Seth Brussaard*, ASML **INVITED**

Extreme Ultraviolet (EUV) lithography is a key enabling technology in semiconductor manufacturing, facilitating the production of the most advanced microchips with smaller feature sizes and greater complexity. Central to this technology is the generation of EUV light through the interaction of plasma and lasers. This presentation explores the critical role of these and other types of plasma present in EUV lithography scanners, highlighting innovations, challenges, and future research directions. The challenges include consistently generating EUV light via laser-produced tin plasma, preventing carbon deposition on optics, and minimizing defects in produced circuits. Addressing these challenges involves multidisciplinary research in plasma-materials interaction, measurements (plasma diagnostics) and control. The ongoing research is aimed at further improving the efficiency and capabilities of EUV lithography systems, driving the development of more advanced and powerful semiconductor devices.

10:00am PS2-FrM-8 Stannane Decomposition and Sticking Coefficient in Extreme Ultraviolet Lithography Environments, *Emily Greene, Nathan Barlett, Jameson Crouse, Eric Mushrush, Alex Shapiro*, University of Illinois; *Niels Braaksma*, ASML; *David Ruzic*, University of Illinois

In extreme ultraviolet (EUV) lithography environments, large quantities of tin are evaporated, leading to the deposition of tin on various chamber surfaces, including collector mirrors. Hydrogen plasma etching is used to remove these deposits, but this process also produces stannane (SnH_4). Since stannane exists in a gaseous state under operational conditions, it can be evacuated from the chamber via a vacuum pump. However, stannane is unstable and often decomposes, causing the redeposition of tin on chamber surfaces. This work aims to experimentally study the decomposition of stannane on EUV-relevant surfaces as a function of temperature. Stannane is synthesized in liquid form through the reaction of lithium aluminum hydride with tin tetrachloride. The liquid stannane is then released into a vacuum chamber containing a temperature-controlled stage equipped with a quartz crystal microbalance (QCM). This setup enables the quantitative determination of the stannane sticking coefficient as a function of surface material and temperature. To analyze surface morphology after stannane exposure, scanning electron microscopy (SEM) is used to image the exposed samples. Additionally, this study seeks to determine the vapor pressure of stannane gas by measuring the pressure of a sealed liquid stannane vessel as it is submerged in chemical slurries of varying

temperatures. By improving the understanding of stannane decomposition, this investigation aims to enhance the maintenance and efficiency of EUV lithographic systems.

Plasma Science and Technology Room 201 ABCD W - Session PS3+TF-FrM

Plasmas and PVD

Moderators: *Erwin Kessels*, Eindhoven University of Technology, the Netherlands, *Scott Walton*, Naval Research Laboratory

10:45am PS3+TF-FrM-11 Self-Regulating Electron Temperature in High-Power Impulse Magnetron Sputtering Discharges and Its Effect on the Metal Ion Escape, *Kateryna Barynova*, University of Iceland; *Nils Brenning*, KTH Royal Institute of Technology, Sweden; *Swetha Suresh Babu*, University of Iceland; *Joel Fischer*, *Daniel Lundin*, Linköping University, Sweden; *Michael A. Raadu*, KTH Royal Institute of Technology, Sweden; *Jon Tomas Gudmundsson*, University of Iceland; *Martin Rudolph*, Leibniz Institute of Surface Engineering (IOM), Germany

We analyze how the primary electron temperature in high-power impulse magnetron sputtering (HiPIMS) depends on the sputtered target. The analysis is based on the experimental discharge data for 7 different target materials, which were modeled using the Ionization Region Model (IRM), a semi-empirical global model for HiPIMS discharges. We observe that the electron heating and collisional cooling processes stabilize after some time into the pulse (20 - 40 μs) reaching a steady state and leading to an almost constant electron temperature; the initial transients in the electron temperature are caused by only small discrepancies in these terms. The underlying mechanism that causes this self-regulation are the rate coefficients for electron impact ionization, which increase monotonically with electron temperature. This leads to a self-balancing mechanism in which an increase in the electron temperature increases the collisional losses of the kinetic energy of electrons because of the higher collisions rate with species in the ionization region. The opposite is true for the decreasing electron temperature. In addition, the steady-state electron temperature depends on the target material and inversely correlates with the self-sputter yield of the target. The species composition in the ionization region shifts from being composed of argon species to target species; and argon has a much higher ionization potential compared to all the studied target materials, so both the ionization and cooling rates substantially increase only at the higher electron temperatures compared to the ionization region composed of target species. This explains the experimentally observed low electron temperature in high self-sputter yield target discharges. Since the mean free path of the sputtered atoms, before being ionized, depends on the electron temperature, we can explain with the IRM results why in metal-rich discharges ionization occurs further away from the target, leading to higher chances of ionized sputtered species to escape to the substrate because the electric field is weaker there. The dominating species in the ionization region, which define the main collisional loss process and the electron temperature, are not identified only by the sputter yield of the target, but by a more complex recycling loop of argon and target species in the ionization region and the rarefaction of argon in front of the target.

11:00am PS3+TF-FrM-12 Comparison of Particle Size and Morphology of Graphene-Like Carbon Grown with and Without Substrate in Atmospheric Pressure Microwave Plasma, *Parker Hays*, *Dhruval Patel*, *Dren Qerimi*, University of Illinois at Urbana-Champaign; *Michael Stowell*, Lyten; *David Ruzic*, University of Illinois at Urbana-Champaign

Graphene-like carbon materials were synthesized on a substrate as well as free-standing using an atmospheric pressure microwave plasma (APP) system. Argon and nitrogen were utilized as carrier gases and methane as the carbon precursor. This study compares the morphological and structural differences of the materials formed under each growth condition.

Free-standing carbon material was collected from the APP using quickly inserted TEM grids at various distances from the microwave insertion point. Carbon was also grown on a temperature-controlled aluminum surface using the same APP system by placement of the aluminum substrate at different distances from the microwave insertion point.

Scanning Electron Microscopy (SEM) images were used to find the particle diameter distributions for each case, showing for both the free-standing carbon and the carbon grown on aluminum that mean primary particle size increased as a function of increased methane flow rate and distance from the microwave insertion point, and decreased with an increase in

microwave forward power. Furthermore, the particle diameter distributions in the free-standing case showed minimal change past the bulk plasma boundary, meaning most of the free-standing graphene growth occurred in the bulk plasma and at the boundary.

Raman spectroscopy was employed to evaluate the structural order and defect density of the carbon materials. The free-standing material exhibited a higher I_D/I_G intensity ratio, suggesting increased disorder and a more amorphous structure compared to substrate-grown samples, which displayed sharper G and 2D peaks indicative of more crystalline graphene. These findings suggest that while substrate-free growth at atmospheric pressure offers a more scalable and simple synthesis route, growth on substrate may yield higher structural quality in the resulting carbon materials.

11:15am PS3+TF-FrM-13 Mass Spectrometric Study of Ar-Diluted Ammonia Borane Plasma for h-BN 2d Film Formation, Takeshi Kitajima, Reiji Kawasaki, Toshiki Nakano, National Defense Academy, Japan

Ammonia borane is used as a relatively safe source of BN for the rapid synthesis of h-BN, an important insulating material¹ in the field of two-dimensional electronics². Ammonia borane plasma attracts attention when aiming at high-speed film formation, and analysis of active species in the plasma is necessary. In this study, active species generated from ammonia borane powder irradiated with Ar plasma were analyzed by mass spectrometry. Parallel plate type 100MHz driven capacitively coupled plasma generated in a high vacuum chamber is used. After placing 0.1 g of ammonia borane (BH_3NH_3) powder on the RF electrode and evacuating, a 10 W glow discharge was formed with an Ar gas flow rate of 30 sccm. A copper sample heated to 800°C was placed downstream, and when BN radicals were supplied at a pressure of 800 Pa, an h-BN atomic film was formed over 30 minutes as shown in the SEM image and Raman spectrum of Fig. 1(a,b). Radical analysis in the downstream was performed with a mass spectrometer at a pressure of 30 Pa. Figure 1(c) shows the difference in the mass spectrum when the plasma is turned on and off. $BNH_5(30)$ is increased by plasma lighting. $O_2(32)$ is produced by plasma irradiation to the chamber wall. Radicals generated from ammonia borane raw material leading to formation of h-BN atomic film are presumed to be BNH_5 generated by decomposition of BH_3NH_3 . Dangling bonds of BNH_5 are thought to generate chemical reaction activity on the substrate. Time dependence of mass signal is shown in Fig. 1(d). The relation of OH and BNH_5 is shown in Fig. 1(e). OH is linearly related to BNH_5 amount and presumed to be the major source of production. Contrarily, O signal is nonlinear to the BNH_5 signal as shown in Fig. 1(f). The consequence of the oxygen related radical exposure will be summarised in the presentation. 1 K.H. Lee, et.al. Nano Letters 12, 714 (2012). 2 L. Song, et.al. Nano Letters 10, 3209 (2010).

11:30am PS3+TF-FrM-14 Automated Deposition Chamber for Functional Dielectrics: Development and Implementation, Stanislav Udovenko, Ian Mercer, Susan Troler-McKinstry, Jon-Paul Maria, Darren Pagan, The Pennsylvania State University

S. A. Udovenko¹, I. Mercer¹, S. Troler-McKinstry¹, J. P. Maria¹ and D. C. Pagan¹

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Automated Deposition Chamber for Functional Dielectrics: Development and Implementation

The dielectric and piezoelectric properties of ferroelectrics make them essential in the fabrication of multilayer ceramic capacitors (MLCCs) and various transducers (such as those used in medical ultrasound, naval sonar, and consumer electronics) [1–3]. Modern devices demand high-quality, reproducible material synthesis, especially in the fabrication of complex multilayer structures where the thickness of individual layers critically affects device performance. In this context, automating the material synthesis process becomes highly beneficial, as it reduces human error, increases repeatability, and improves overall efficiency. However, there is currently a disconnect between university-based materials design and synthesis which is primarily an analog process and large-scale automated manufacturing found in industry.

This project focuses on developing a framework for digitizing and automating functional ferroelectric synthesis in a university setting. Our demonstration case is the sputtering of ferroelectric films within a vacuum deposition chamber. In the initial stage, a data acquisition and controller system was designed and installed on deposition chamber automated for

doped AlN. Next, LabVIEW-based software was developed to acquire data from all electronic units of the chamber—including sputter cathode power supplies, mass flow controllers, temperature controllers, and vacuum pumps. Following software development, control functionality was implemented, enabling the system to send control commands to all electronic units while simultaneously logging process parameters in real time. Additionally, Python scripts were developed to convert deposition recipes—originally created in Microsoft Excel—into system control routines, easing use by non-experts.

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Bold page numbers indicate presenter

— A —

A. I. Elgarhy, Mahmoud: PS3-TuA-12, 14
 Abdelghany, Muhammad: PS-ThP-22, **43**
 Abdulgatov, Aziz: AP+PS+TF-WeA-5, **23**
 Abel, Kate: AP+PS+TF-WeA-3, 22
 Abuyazid, Nabil: PS2-TuA-10, 13
 Agarwal, Prawal: AP+PS+TF-ThA-4, 37
 Agarwal, Sumit: PS-TuM-14, **10**; PS-TuM-17, 11; PS-WeM-15, 20
 Akter, Tanjina: PS-ThP-25, **43**
 Alberi, Kirstin:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-5, **7**
 Alsaed, Omar: PS-MoA-3, **4**
 Altieri, Nicholas: AP+EM+PS+TF-FrM-8, 49
 Ammons, Keanu: PS-ThP-34, 45
 An, Jae-Seok: AP+PS+TF-WeM-1, 16
 Ando, Yusuke: PS1-FrM-1, **50**
 Andrianov, Nikolai: AP+PS+TF-WeA-10, **24**
 Aoyagi, Chikashi: PS1-TuA-1, 13
 Arkalgud, Sitaram: PS1-TuA-1, 13
 Armini, Silvia: AP+EM+PS+TF-FrM-10, **49**
 Aronson, Benjamin:
 EM1+AP+CPS+MS+PS+SM+TF-WeA-1, 26;
 EM1+CPS+MS+PS+SM+TF-TuA-2, **12**
 Arslanbekov, Robert: PS+AIML-ThA-5, 39
— B —
 Bai, Keun Hee: AP+PS+TF-WeA-14, 25
 Baksi, Merve:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-8, **8**
 Bale, Raja Sekhar: EM1+CPS+MS+PS+SM+TF-TuA-1, **12**
 Balliana, Eleonora: AP+PS+TF-ThA-1, 37
 Banerjee, Parag: AP+PS+TF-ThA-2, 37;
 AP+PS+TF-WeM-8, 17
 Barber, John: EM1+AP+CPS+MS+PS+SM+TF-WeA-2, 26
 Barlett, Nathan: PS2-FrM-8, 51
 Barry, Sean: AP+PS+TF-ThA-8, **38**
 Barsukov, Yuri: PS3-WeA-9, **28**
 Bartlett, Nathan: PS-ThP-27, 44; PS-ThP-28, 44; PS-ThP-29, **44**
 Barynova, Kateryna: PS3+TF-FrM-11, **51**
 Bejger, Gerald:
 EM1+AP+CPS+MS+PS+SM+TF-WeA-2, 26
 Beji, Hiba: PS-TuM-3, 9
 Benotmane, Khatia: PS1-WeA-1, 27
 Bent, Stacey: AP+EM+PS+TF-FrM-5, **48**
 Bentz, Brian: PS-MoA-3, 4
 Bepari, Sujoy: PS-WeM-7, 20
 Bera, Kallol: PS+AIML-ThA-1, 39; PS-MoA-1, 4; PS-MoA-14, 6; PS-MoA-4, 4; PS-MoA-7, 5; PS-MoA-9, 5; PS-ThP-35, **46**
 Berriel, Novia: AP+PS+TF-WeM-8, 17
 Bevan, Malcolm: PS-ThP-40, 47
 Bezard, Philippe: PS2-TuA-11, 13; PS-MoM-14, 3
 Bézard, Philippe: PS-MoM-13, 3
 Bhuian, Ali: PS+AIML-ThA-4, 39
 Bhuva, Montu: PS-MoA-13, **6**
 Bielinski, Ashley: AP+PS+TF-ThA-5, **37**
 Biolsi, Peter: PS-MoM-10, 2
 Bissell, Eric: AP+PS+TF-ThA-2, **37**
 Blakeney, Kyle: AP+EM+PS+TF-FrM-12, **49**
 Blanco Carballo, Victor M.: PS-MoM-14, 3
 Boixaderas, Christelle: PS-TuM-1, 8; PS-TuM-2, 8
 Bonvalot, Marcelline: AP+PS+TF-WeA-13, 24
 Boris, David: AP+PS+TF-WeM-17, 19; PS3-WeA-13, 29; PS-ThP-16, 42; PS-ThP-36, 46
 Bowman, Chris: PS-ThP-23, 43; PS-ThP-24, 43
 Boyce, Gillian: AP+PS+TF-ThA-1, **37**
 Braaksma, Niels: PS2-FrM-8, 51; PS-ThP-28, 44
 Brenning, Nils: PS3+TF-FrM-11, 51
 Author Index

Brussaard, Seth: PS2-FrM-6, **51**
 Budach, Michael: PS-MoM-4, 1
 Bufi, Giuseppe Libero: PS-ThP-33, **45**
 Buseyne, Daan:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, 7
 Butkus, Brian: AP+PS+TF-ThA-2, 37
— C —
 Cahen, David:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-7, 7
 Caielli, Francesca: PS-WeM-5, 19
 Calderon, Sebastian:
 EM1+CPS+MS+PS+SM+TF-TuA-3, 12
 Calleri, Loren: PS-ThP-21, 43
 Camacho, Vitor: PS-WeM-1, 19; PS-WeM-2, 19
 Camata, Renato: PS+AIML-ThA-5, **39**
 Cardinaud, Christophe: PS-TuM-3, **9**
 Caribe, Zuriel: AP+PS+TF-WeM-6, 17
 Carroll, Nicholas: AP+AS+EL+EM+PS+TF-ThM-7, **32**; AP+EM+PS+TF-FrM-7, 49
 Caruso, Anthony: EM1+CPS+MS+PS+SM+TF-TuA-1, 12
 Cary, John: PS3-WeA-15, 29
 Cascales, David: PS1-WeA-1, 27
 Cavanagh, Andrew: PS2-ThM-13, 35
 Cemin, Felipe: PS-TuM-3, 9
 Chae, Heeyeop: PS3-TuA-13, 14; PS-ThP-38, 46
 Chakraborty, Shashwata: PS-WeM-7, 20
 Chan, Kelvin: PS1-ThM-8, 35
 Chang, Jane: AP+EM+PS+TF-FrM-8, 49
 Chang, Jane P.: AP+PS+TF-WeA-15, 25
 Chen, Guohai: PS1-FrM-2, **50**
 Chen, Ivy: AP+PS+TF-WeA-9, 23
 Chen, Jiun-Ruey: AP+AS+EL+EM+PS+TF-ThM-8, 32
 Chen, Timothy: PS1-ThM-8, 35
 Chen, Yi: AP+EM+PS+TF-FrM-8, **49**
 Chen, Yuaning: PS-ThP-40, 47
 Cheng, Zhongyu: PS-ThP-18, 42
 Chevolleau, Thierry: PS-TuM-1, 8
 Chiang, Wei-Hung: PS-WeM-5, 19
 ChinWook, Chung: PS-ThP-32, 45
 Cho, Chulhee: PS1-ThM-1, 34; PS1-ThM-2, 34; PS1-ThM-5, 34
 Cho, Daniel: AP+EM+PS+TF-FrM-8, 49
 Cho, Nam Il: PS-TuM-7, 10
 Choi, Boyun: AP+EM+PS+TF-FrM-4, **48**
 Choi, Byeongyeop: PS1-ThM-1, 34; PS1-ThM-2, 34; PS1-ThM-5, 34
 Choi, Hyeonjin: PS3-TuA-13, 14
 Choi, Minsu: PS1-ThM-1, 34; PS1-ThM-2, 34; PS1-ThM-5, 34
 Choi, Tag: PS2-ThM-14, **35**
 Choi, Yong Kyu:
 EM1+AP+CPS+MS+PS+SM+TF-WeA-1, **26**
 Chopra, Nirbhav: PS1-ThM-6, 34; PS-ThP-12, **41**
 Chowdhary, Nimarta: AP+PS+TF-WeM-3, 16
 Chowdhury, Rajib: PS-MoM-12, **2**
 Chowdhury, Tanzia: AP+PS+TF-WeM-1, 16
 Chowrira, Bhavishya: PS-MoM-14, 3
 Chung, Chin Wook: PS-ThP-5, 40
 Chung, Chin-Wook: PS3-TuA-15, 15; PS-ThP-19, 43
 Chung, Sang-Jin: PS-MoM-11, **2**
 Colleran, Troy: AP+PS+TF-WeA-2, 22
 Conley Jr., John: AP+PS+TF-WeM-4, 16
 Crouse, Jameson: PS2-FrM-8, 51; PS-ThP-27, 44; PS-ThP-28, **44**; PS-ThP-29, 44
 Crowley, Brendan: PS-ThP-34, 45
 Currie, Taylor: AP+PS+TF-WeM-8, 17
— D —
 Dalton, Jeremie: AP+EM+PS+TF-FrM-12, 49

Daly, Gregory: PS-MoA-13, 6
 Dao, Thang: AP+PS+TF-WeA-10, 24
 Das, Shubhankar: PS-MoM-14, 3
 Das, Tridip: PS-WeM-7, 20
 Davda, Kirtan: PS-ThP-34, 45
 De Gendt, Stefan: PS2-TuA-11, 13
 de Marneffe, Jean-François: AP+PS+TF-WeA-15, 25
 de Rooij, Raoul: PS2-FrM-5, 50
 Dearing, Matthew T.: AP+AS+EL+EM+PS+TF-ThM-6, 31
 Debastiani, Benjamin:
 EM1+CPS+MS+PS+SM+TF-TuA-3, 12
 Decoster, Stefan: PS-MoM-14, 3
 Dedrick, James: PS-MoA-13, 6
 del Hoyo, Javier: PS-ThP-16, 42
 Delie, Gilles:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, 7
 Demaude, Annaelle: PS-MoM-14, 3
 Denchy, Mike: AP+PS+TF-ThA-4, 37
 Deng, Yubin: AP+PS+TF-WeM-14, **18**
 deVilliers, Anton: AP+PS+TF-WeM-6, 17
 Dickey, Elizabeth: EM1+CPS+MS+PS+SM+TF-TuA-2, 12; EM1+CPS+MS+PS+SM+TF-TuA-3, 12
 Dickey, Michael: AP+PS+TF-WeM-2, 16
 Dimova, Dexter: AP+PS+TF-ThA-8, 38
 Donnelly, Vincent: AP+PS+TF-WeA-12, 24; PS3-TuA-12, **14**
 Draney, Jack: AP+AS+EL+EM+PS+TF-ThM-5, **31**
 Dren, Qerimi: PS-ThP-26, 44
 Dryzhakov, Bogdan:
 EM1+CPS+MS+PS+SM+TF-TuA-2, 12
 Dubois, Jérôme: PS-TuM-2, 8
 Duffield, Micah: AP+PS+TF-WeA-1, 22; AP+PS+TF-WeA-3, 22
— E —
 Edinger, Klaus: PS-MoM-4, 1
 Egly, Matt: PS2-ThM-14, 35
 Elam, Jeffrey W.: AP+AS+EL+EM+PS+TF-ThM-6, 31
 Elgarhy, Mahmoud A. I.: AP+PS+TF-WeA-12, 24
 Ellis, Daniel: PS2-TuA-10, **13**
 Ellis, James: PS1-ThM-3, 34; PS-MoA-13, 6; PS-MoA-16, 6
 Elshaer, Adham:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, 7
 Engeln, Richard: PS2-ThM-16, 36
 Engstrom, James: AP+EM+PS+TF-FrM-13, **49**
 Eoh, Hyeon Joon: PS-ThP-9, **41**
 Eriguchi, Koji: PS-MoM-1, **1**
 Eslamisaray, Mohammadali: PS2-WeA-5, 28
— F —
 Farr, Jon: AP+PS+TF-WeA-10, 24
 Fathzadeh, Atefeh: PS2-TuA-11, **13**
 Ferraris, Sergio: PS-ThP-28, 44
 Fischer, Joel: PS3+TF-FrM-11, 51
 Fisher, Ellen R.: PS1-FrM-3, **50**
 Fletcher, Ivan: AP+PS+TF-WeM-6, 17
 Fontaine, Benjamin: PS-TuM-2, **8**
 Friedrichs, Niklas: PS-MoA-1, 4
 Fuchs, Ehud: AP+PS+TF-ThA-6, 38
 Fuerst, Thomas: PS-WeM-1, 19; PS-WeM-2, 19
 Fulford, Jim: AP+PS+TF-WeM-6, 17
 Futaba, Don: PS1-FrM-2, 50
— G —
 Ganta, Sathya: PS-MoA-7, **5**; PS-ThP-35, 46
 Gao, Jeffrey: AP+EM+PS+TF-FrM-13, 49
 Gaucher, François: PS1-WeA-1, 27
 Gaume, Romain: AP+PS+TF-ThA-2, 37

Author Index

George, Steven: AP+PS+TF-ThA-9, 38;
 AP+PS+TF-WeA-1, **22**; AP+PS+TF-WeA-2,
 22; AP+PS+TF-WeA-3, 22; AP+PS+TF-WeA-
 5, 23; PS2-ThM-13, 35
 Gheeraert, Etienne: AP+PS+TF-WeA-13, 24
 Gil, Hong Seong: PS-MoM-5, **1**; PS-ThP-31,
 45; PS-ThP-4, 40; PS-ThP-8, 41
 Gillmore, Emily: PS2-WeA-5, 28
 Girard, Aurélie: PS-TuM-3, 9
 Go, David: PS-ThP-18, 42
 Gobil, Yveline: PS1-WeA-1, 27
 Goddard, William: PS-WeM-7, 20
 Gokhale, Vikrant J.: AP+PS+TF-WeM-15, 18
 Goodin, Aunic: PS-WeM-7, **20**
 Gougousi, Theodosia: AP+PS+TF-WeM-3, **16**
 Gouraud, Pascal: PS-TuM-2, 8
 Granat, Jack: PS-ThP-29, 44
 Graugnard, Elton: AP+AS+EL+EM+PS+TF-
 ThM-4, 31
 Graves, David: AP+AS+EL+EM+PS+TF-ThM-3,
 31; AP+AS+EL+EM+PS+TF-ThM-5, 31; PS2-
 WeA-6, 28
 Greene, Emily: PS2-FrM-8, **51**; PS-ThP-27, 44;
 PS-ThP-28, 44; PS-ThP-29, 44
 Greer, Frank: AP+PS+TF-WeA-9, 23
 Grenouillet, Laurent: PS-TuM-1, 8
 Griffiths, Matthew: AP+EM+PS+TF-FrM-12,
 49
 Grzeskowiak, Steven: PS-MoM-6, 2
 Gu, Bonwook: AP+PS+TF-WeM-7, 17
 Guaitella, Olivier: PS-WeM-5, 19
 Gudmundsson, Jon Tomas: PS3+TF-FrM-11,
 51
 Gui, Yifan: PS-MoA-12, **5**; PS-TuM-5, 9
 Gunay, Ece: EM1+CPS+MS+PS+SM+TF-TuA-2,
 12
 Gupta, Shiva: PS+AIML-ThA-5, 39
 Guttmann, Jan: PS-MoA-1, 4; PS-MoA-14, 6
— H —
 Ha, Dae Yeon: PS-ThP-31, 45; PS-ThP-8, 41
 Ha, Seokhyeon: PS-ThP-19, **43**
 Habib, Matias: PS-ThP-29, 44
 Hackett, Aaron: PS2-ThM-14, 35
 Haglund, Jessica: AP+PS+TF-WeM-4, **16**
 Hamaguchi, Satoshi: PS-ThP-14, 42
 Han, Hyewon: PS-ThP-11, 41
 Hanna, Avery:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-8, 8
 Hao, Qinzhen: AP+PS+TF-WeA-12, 24; PS3-
 TuA-12, 14
 Haque, Jami Md Ehsanul: PS1-ThM-2, 34
 Haque, Jami MD Ehsanul: PS1-ThM-1, 34
 Harris, Ben: PS-MoA-16, 6
 Harris, Sumner: PS+AIML-ThA-5, 39
 Harzenetter, Steffen: AP+PS+TF-WeA-10, 24
 Hassall, Geoff: PS-MoA-13, 6; PS-MoA-16, 6
 Hata, Kenji: PS1-FrM-2, 50
 Hausmann, Dennis M.:
 AP+AS+EL+EM+PS+TF-ThM-16, 33
 Hays, Parker: PS3+TF-FrM-12, **51**
 Held, Julian: PS2-ThM-16, 36
 Henry, M. David:
 EM1+AP+CPS+MS+PS+SM+TF-WeA-3, 26;
 EM1+AP+CPS+MS+PS+SM+TF-WeA-4, 26
 Heo, Subin: AP+PS+TF-WeA-6, 23
 Herr, Anna:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, 7
 Herr, Quentin:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, 7
 Herschberg, Andrew: PS-ThP-28, 44; PS-ThP-
 29, 44
 Herschberg, Andrew C: PS-ThP-27, **44**
 Hill, Lewis: PS+AIML-ThA-3, 39
 Hinshelwood, Michael: PS-MoM-4, **1**
 Hoang, John: AP+EM+PS+TF-FrM-8, 49

Hodges, Blake:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, 7
 Holler, Logan: PS-ThP-26, **44**
 Holsgrove, Kristina:
 EM1+AP+CPS+MS+PS+SM+TF-WeA-1, 26
 Hong, Jong Woo: PS-ThP-4, 40; PS-ThP-6, 40;
 PS-ThP-7, 40; PS-TuM-7, **10**
 Hood, Zachary: AP+PS+TF-WeM-16, 18
 Hopkins, Janet: PS+AIML-ThA-3, 39
 Hori, Masaru: AP+PS+TF-WeA-16, 25;
 AP+PS+TF-WeA-4, 22; PS-TuM-15, 10; PS-
 TuM-8, 10
 Hossain, Md Tanzid: PS-TuM-14, 10; PS-TuM-
 17, **11**
 Hossain, Mohammad Sazzad: PS-ThP-34, **45**
 Houben, Ralph: PS2-ThM-16, **36**
 Hsiao, Shih-Nan: PS-TuM-15, **10**
 Huang, Chenyao: PS-MoA-12, 5; PS-TuM-5, **9**
 Huang, Shuo: PS+AIML-ThA-3, 39
 Huard, Chad: PS-TuM-13, 10
 Huard, Chad M.: PS+AIML-ThA-3, **39**
 Hues, John D.: AP+AS+EL+EM+PS+TF-ThM-4,
 31
 Hues, Steven M.: AP+AS+EL+EM+PS+TF-
 ThM-4, 31
 Huet, Benjamin:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, 7
 Hufnagel, Caroline: PS-WeM-1, **19**; PS-WeM-
 2, 19
 Hwang, Gyeong: AP+AS+EL+EM+PS+TF-ThM-
 14, 33
 Hyungdong, Kim: PS-ThP-32, **45**
 HyunHo, Nahm: PS-ThP-32, 45
— I —
 Ibrahim, Seifallah:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, 7
 Ihlefeld, Jon: EM1+AP+CPS+MS+PS+SM+TF-
 WeA-2, 26; EM1+AP+CPS+MS+PS+SM+TF-
 WeA-3, 26; EM1+AP+CPS+MS+PS+SM+TF-
 WeA-4, 26; EM1+CPS+MS+PS+SM+TF-TuA-
 2, 12
 Ihlefeld, Jon F.:
 EM1+AP+CPS+MS+PS+SM+TF-WeA-1, 26
 Iijima, Yuki: PS-TuM-15, 10
 Im, Yeon Ho: PS-ThP-15, 42
 Imai, Yusuke: PS-TuM-15, 10
 Ishihara, Takuya: PS-ThP-14, **42**
 Ishii, Takayuki: PS1-TuA-1, 13
 Ishikawa, Dai: AP+PS+TF-WeA-16, 25
 Ishikawa, Kenji: AP+PS+TF-WeA-16, 25;
 AP+PS+TF-WeA-4, 22; PS1-FrM-1, 50; PS-
 TuM-8, **10**
 Izawa, Masaru: AP+PS+TF-WeA-16, 25;
 AP+PS+TF-WeA-4, 22
— J —
 J. Van Bael, Margriet:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, 7
 Jami, Ehsanul Haque: PS1-ThM-5, 34
 Janda, Mario: PS-WeM-5, 19
 Jang, Jinhyeok: PS1-ThM-1, 34
 Jang, SeonHee: PS-MoM-12, 2
 Jang, Suyoung: PS-ThP-37, 46
 Jang, Yun Jong: PS-MoM-5, 1; PS-ThP-31, **45**;
 PS-ThP-8, 41
 Jaszewski, Samantha:
 EM1+AP+CPS+MS+PS+SM+TF-WeA-3, 26;
 EM1+AP+CPS+MS+PS+SM+TF-WeA-4, **26**
 Jeckell, Zachary: PS2-ThM-14, 35
 Jenkins, Thomas: PS3-WeA-15, 29
 Jensen, Brian: PS2-WeA-6, **28**
 Jensen, James: AP+EM+PS+TF-FrM-13, 49
 Jeon, Nari: AP+EM+PS+TF-FrM-4, 48
 Jeong, Jun Won: PS-ThP-7, **40**
 Jeong, Sun Jae: PS-ThP-30, 45
 Jeong, Sunjae: PS-ThP-11, **41**

Jeong, Wonnyoung: PS1-ThM-1, 34; PS1-
 ThM-2, 34; PS1-ThM-5, 34
 Jeong, Yeong Jae: PS-ThP-5, 40
 Jeong, Yeongjae: PS3-TuA-15, **15**
 Johnson, Johnathon: AP+PS+TF-WeM-8, 17
 Johnson, Michael: PS3-WeA-13, 29; PS-ThP-
 16, 42; PS-ThP-36, 46
 Jones, Jessica: AP+PS+TF-WeM-16, **18**
 Jones, Jessica C.: AP+AS+EL+EM+PS+TF-ThM-
 6, 31
 Joy, Nicholas: PS-MoM-16, 3
 Jung, Haegeon: PS-ThP-38, **46**
 Jung, Sun Kyu: AP+PS+TF-WeA-6, 23
 Jung, Un Jae: PS-ThP-5, **40**
 Junige, Marcel: AP+PS+TF-WeA-1, 22
 Jurca, Titel: AP+PS+TF-WeM-8, 17
 Jurczyk, Brian: PS2-TuA-10, 13
— K —
 Kaganovich, Igor: PS3-WeA-11, **29**
 Kahn, Antoine:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-7, 7
 Kalas, Benjamin: AP+PS+TF-WeA-10, 24
 Kallenberg, Evan: PS-ThP-34, 45
 Kamiyama, Brandon: PS2-WeA-5, **28**
 Kampitakis, Viktor: PS-MoM-14, 3
 Kanfer, David S.: PS-ThP-25, 43
 Kang, Hojun: PS-ThP-14, 42
 Kang, Jieun: PS-ThP-11, 41
 Kang, Song-Yung: PS3-TuA-12, 14
 Kang, Xin: AP+PS+TF-WeM-8, 17
 Kang, Youngho: AP+PS+TF-WeM-7, 17
 Karahashi, Kazuhiro: PS-ThP-14, 42
 Karpatne, Anand: PS3-WeA-14, 29
 Katakam, Shravana Kumar: PS1-TuA-3, 13;
 PS-MoM-16, **3**
 Kawasaki, Reiji: PS3+TF-FrM-13, 52
 Kaya, Andac Yagiz: PS-WeM-17, 21
 Kelley, Kyle: EM1+CPS+MS+PS+SM+TF-TuA-2,
 12
 Kenney, Jason: PS3-WeA-12, 29; PS-MoA-1, 4
 Kerr, Sean: PS-ThP-18, 42
 Kessels, Erwin: AP+PS+TF-WeM-5, **17**; PS1-
 ThM-3, 34; PS2-ThM-16, 36
 Kessels, Wilhelmus M. M.: AP+EM+PS+TF-
 FrM-1, 48
 Khaji, Maryam: AP+PS+TF-WeA-12, **24**
 Khrabrov, Alexander: PS3-WeA-11, 29
 Kihara, Yoshihide: PS-TuM-15, 10
 Kim, Beomseok: AP+PS+TF-WeA-2, 22
 Kim, Chan Ho: PS-ThP-6, **40**
 Kim, Changyu: AP+PS+TF-WeM-1, 16
 Kim, Dohyeon: PS-ThP-37, 46
 Kim, Eun Koo: PS-ThP-2, **40**
 Kim, Heejung: PS3-TuA-12, 14
 Kim, Hye-Lee: AP+PS+TF-WeM-1, 16
 Kim, Hyeongkeun: AP+PS+TF-WeM-7, 17
 Kim, Hyunmi: AP+PS+TF-WeM-7, 17
 Kim, Jieun: AP+PS+TF-WeA-6, **23**
 Kim, Jimin: PS-ThP-11, 41
 Kim, Jin Ha: AP+PS+TF-WeA-6, 23
 Kim, Jinmyeong: PS3-TuA-13, 14
 Kim, Ju Won: PS-ThP-15, **42**
 Kim, Kyongnam: PS-ThP-37, **46**
 Kim, Kyoung Chan: PS-ThP-31, 45; PS-ThP-8,
41
 Kim, Min Seok: PS-ThP-5, 40
 Kim, Minseok: PS-ThP-19, 43
 Kim, Namgun: PS3-TuA-13, 14
 Kim, Okhyeon: AP+PS+TF-WeM-1, **16**
 Kim, Sang-Hoon: AP+PS+TF-WeA-6, 23
 Kim, Seoyeon: AP+PS+TF-WeM-2, 16
 Kim, Sijun: PS1-ThM-2, 34; PS1-ThM-5, 34
 Kim, Sungjoon: AP+AS+EL+EM+PS+TF-ThM-
 6, 31
 Kim, Sungyeol: PS1-ThM-7, 35

Author Index

Kim, Wonjoong: AP+PS+TF-WeM-7, **17**
 Kintzer, Josh: AP+PS+TF-ThA-4, 37
 Kitajima, Takeshi: PS3+TF-FrM-13, **52**; PS-ThP-13, **41**
 Knoops, Harm: PS1-ThM-3, 34
 Ko, Youngju: PS3-TuA-13, **14**
 Kolobov, Vladimir: PS-MoA-5, **4**
 Korolov, Ihor: PS-MoA-1, 4; PS-MoA-14, 6
 Kostogiannes, Alexandros: AP+PS+TF-ThA-2, 37
 Kothnur, Prashanth: PS3-TuA-14, 14
 Kozuch, Sandra: PS1-WeA-3, **27**
 Krogstad, Jessica: PS2-TuA-10, 13
 Kruger, Scott: PS3-WeA-15, 29
 Kuila, Debasish: PS-WeM-7, 20
 Kulshreshtha, Prashant: PS-ThP-35, 46
 Kumar, Amit: EM1+AP+CPS+MS+PS+SM+TF-WeA-1, 26
 Kumar, Prabhat: PS-TuM-14, 10; PS-TuM-17, 11
 Kurniawan, Darwin: PS-WeM-5, 19
 Kurushima, Takumi: PS-TuM-8, 10
 Kushner, Mark: PS3-WeA-13, 29
 Kushner, Mark J.: AP+PS+TF-WeA-12, 24; PS-MoA-12, 5; PS-TuM-16, 11; PS-TuM-5, 9
 Kuwahara, Kenichi: PS-MoM-3, 1
 Kwak, Byungha: AP+PS+TF-WeM-7, 17
— L —
 Laggner, Florian: PS-ThP-34, 45
 Lagunas Vargas, Francisco: AP+PS+TF-WeM-16, 18
 Lalor, James: PS2-WeA-4, 28
 Lam, Nicolas: EM1+AP+CPS+MS+PS+SM+TF-WeA-2, **26**
 Lass, Steve: AP+PS+TF-ThA-2, 37
 Lazzarino, Frederic: PS-MoM-14, 3
 Le Pape, Thomas: PS-TuM-3, 9
 Lee, Dongki: PS1-ThM-1, 34; PS1-ThM-2, 34; PS1-ThM-5, 34
 Lee, Eunseo: PS-ThP-39, **46**
 Lee, Geuntak: PS3-WeA-12, 29
 Lee, Hae June: PS-ThP-39, 46
 Lee, Han-Bo-Ram: AP+PS+TF-ThA-3, 37; AP+PS+TF-WeM-7, 17
 Lee, Hyunjae: AP+PS+TF-WeA-12, 24; PS-TuM-16, 11
 Lee, Isak: PS1-ThM-1, **34**; PS1-ThM-2, 34; PS1-ThM-5, 34
 Lee, Joe: PS-MoM-16, 3
 Lee, Sanghyun: AP+PS+TF-WeA-14, **25**
 Lee, Seong Hyun: AP+PS+TF-WeA-6, 23
 Lee, Won-Jun: AP+PS+TF-WeM-1, 16
 Lee, Woobeen: PS1-ThM-1, 34; PS1-ThM-2, 34; PS1-ThM-5, 34
 Lee, Youngseok: PS1-ThM-2, 34
 Lefevre, Scott: PS1-TuA-1, 13
 Lenox, Megan:
 EM1+AP+CPS+MS+PS+SM+TF-WeA-1, 26;
 EM1+AP+CPS+MS+PS+SM+TF-WeA-2, 26;
 EM1+AP+CPS+MS+PS+SM+TF-WeA-3, **26**;
 EM1+AP+CPS+MS+PS+SM+TF-WeA-4, 26
 Leong, Icelene: AP+AS+EL+EM+PS+TF-ThM-4, 31
 Levine, Igal:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-7, 7
 Levko, Dmitry: PS3-WeA-14, 29; PS-MoA-11, **5**
 Li, Chao: PS-WeM-2, 19
 Li, Chen: AP+PS+TF-WeA-2, **22**
 Li, Hu: AP+AS+EL+EM+PS+TF-ThM-14, 33; AP+AS+EL+EM+PS+TF-ThM-15, 33; PS1-FrM-1, 50; PS-MoA-15, **6**
 Li, Junjie: AP+PS+TF-ThA-7, 38
 Li, Shi: AP+PS+TF-WeM-16, 18
 Lie, Fee Li: PS1-TuA-3, **13**

Lietz, Amanda: PS-MoA-3, 4; PS-ThP-34, 45
 Lietz, Amanda M.: PS-MoA-8, 5
 Lill, Thorsten: PS3-WeA-9, 28; PS-TuM-14, 10; PS-TuM-17, 11; PS-WeM-15, 20
 Lim, Hanjin: AP+PS+TF-WeA-2, 22
 Lim, Meehyun: PS1-ThM-7, 35
 Liston, Connor: PS-ThP-24, 43
 Litwin, Peter: AP+PS+TF-WeM-17, 19
 Litwin, Peter M.: AP+PS+TF-WeM-15, 18
 Liu, Eric: PS-MoM-6, 2
 Lodeiro, Lucas: AP+AS+EL+EM+PS+TF-ThM-13, 32; AP+AS+EL+EM+PS+TF-ThM-16, **33**
 Lu, Ming: PS-TuM-6, 9
 Lu, Ting: PS2-WeA-5, 28
 Luan, Pingshan: PS-MoM-10, 2; PS-MoM-11, 2
 Lundin, Daniel: PS3+TF-FrM-11, 51
 Luo, Han: PS3-WeA-12, 29; PS-MoA-1, 4; PS-MoA-14, 6
— M —
 Ma, Zhihao: PS-ThP-40, **47**
 Maas, Joost: AP+EM+PS+TF-FrM-2, **48**
 Maas, Joost F. W.: AP+AS+EL+EM+PS+TF-ThM-13, 32; AP+EM+PS+TF-FrM-1, 48
 Maccio, Bart: AP+PS+TF-WeM-5, 17; PS2-ThM-16, 36
 Mackus, Adriaan: AP+AS+EL+EM+PS+TF-ThM-8, 32
 Mackus, Adriaan J. M.:
 AP+AS+EL+EM+PS+TF-ThM-13, 32;
 AP+AS+EL+EM+PS+TF-ThM-16, 33;
 AP+EM+PS+TF-FrM-1, 48
 Mackus, Adrie: AP+EM+PS+TF-FrM-2, 48
 Maeda, Kenji: AP+PS+TF-WeA-4, 22
 Mahapatra, Jagannath: PS+AIML-ThA-4, 39
 Main, Daniel: PS3-WeA-15, **29**
 Makoto, Sekine: PS-TuM-15, 10
 Mamonuru, Meenakshi: PS-MoA-3, 4
 Mandia, David: AP+EM+PS+TF-FrM-12, 49
 Mani, Radhika: PS3-WeA-14, 29
 Mannequin, Cédric: PS-TuM-3, 9
 Manukyan, Lucy: PS-ThP-41, **47**
 Marbach, Hubertus: PS-MoM-4, 1
 Margavio, Hannah: AP+PS+TF-WeA-11, **24**
 Maria, Jon-Paul: EM1+CPS+MS+PS+SM+TF-TuA-2, 12; EM1+CPS+MS+PS+SM+TF-TuA-3, 12; PS3+TF-FrM-14, 52
 Marion, Jason: PS-MoM-10, 2
 Martinez, Eugénie: PS1-WeA-1, 27
 Matsui, Miyako: PS-MoM-3, **1**
 Matsukuma, Masaaki: PS1-FrM-1, 50; PS-MoA-15, 6
 Mattingly, John: PS1-ThM-7, 35
 Mauri-Newell, Blaine: AP+PS+TF-ThA-2, 37
 Mays, Ebony: PS+AIML-ThA-4, 39
 Mazzeo, Arthur: PS-ThP-34, 45
 Mbouja Signe, Tatiana: PS-TuM-3, 9
 McDonnell, Stephen J.:
 EM1+AP+CPS+MS+PS+SM+TF-WeA-1, 26
 McElwee-White, Lisa: AP+PS+TF-WeM-8, 17
 Mcnealy-James, Terrick: AP+PS+TF-ThA-2, 37
 McNealy-James, Terrick: AP+PS+TF-WeM-8, **17**
 Melikyan, Harutyun: PS+AIML-ThA-4, **39**
 Melnik, Paul: PS1-ThM-7, 35; PS-ThP-23, 43; PS-ThP-24, 43
 Mercer, Ian: EM1+CPS+MS+PS+SM+TF-TuA-2, 12; EM1+CPS+MS+PS+SM+TF-TuA-3, **12**; PS3+TF-FrM-14, 52
 Merckx, Marc: AP+AS+EL+EM+PS+TF-ThM-8, 32; AP+EM+PS+TF-FrM-2, 48
 Merckx, Marc J. M.: AP+AS+EL+EM+PS+TF-ThM-13, 32; AP+AS+EL+EM+PS+TF-ThM-16, 33; AP+EM+PS+TF-FrM-1, 48
 Mette, Gerson: PS-MoM-4, 1

Mettler, Jeremy: AP+PS+TF-WeA-12, 24; PS3-TuA-12, 14
 Meyer, Mackenzie: PS3-WeA-13, **29**; PS-ThP-36, **46**
 Meyer, Makenzie: PS-ThP-16, 42
 Michaud, Vincent: PS-TuM-1, **8**
 Mignot, Yann: PS1-TuA-3, 13; PS-MoM-16, 3
 Miles, Max: PS2-ThM-15, **36**
 Miller, Kenneth E.: PS-ThP-23, 43; PS-ThP-24, 43
 Milosavljevic, Vladimir: PS2-WeA-4, **28**
 Mimura, Yuji: PS1-TuA-1, 13
 Min, Yeongmin: AP+PS+TF-ThA-3, **37**
 Mine, Yousuke: PS1-TuA-1, 13
 Minnich, Austin: AP+PS+TF-WeA-9, **23**
 Miura, Makoto: PS-MoM-3, 1
 Miyake, Machiko: PS-ThP-13, 41
 Mohammad, Adnan: AP+AS+EL+EM+PS+TF-ThM-6, 31
 Mohr, Sebastian: PS-ThP-41, 47
 Moore, Braden: PS-ThP-21, **43**
 Mora, Ayelen: AP+PS+TF-ThA-2, 37
 Motoyama, Koichi: PS-MoM-16, 3
 Mukhopadhyay, Partha: AP+PS+TF-WeM-6, **17**
 Muñoz, Pascual: PS-ThP-33, 45
 Murphy, John: PS-ThP-16, 42
 Mushrush, Eric: PS2-FrM-8, 51
— N —
 Na, Jeong-Seok: AP+EM+PS+TF-FrM-12, 49
 Nagai, Souga: AP+PS+TF-WeM-13, 17; AP+PS+TF-WeM-14, 18
 Nahm, Hyeonho: PS-ThP-19, 43
 Nakano, Takeo: PS-MoA-15, 6
 Nakano, Toshiki: PS3+TF-FrM-13, 52; PS-ThP-13, 41
 Nakaya, Michiko: PS1-TuA-1, 13
 Nam, Chang-Yong: PS-TuM-6, 9
 Nam, Sang Ki: AP+PS+TF-WeA-12, 24; PS3-TuA-12, 14; PS-TuM-16, 11
 Nam, Taewook: AP+PS+TF-ThA-9, **38**
 Neefs, Alex: AP+PS+TF-WeM-5, 17
 Nepal, Neeraj: AP+PS+TF-WeM-15, 18; AP+PS+TF-WeM-17, 19
 Nesterenko, Iurii: AP+PS+TF-WeA-10, 24
 Netzband, Christopher: PS1-TuA-1, 13
 Ngan, Zachariah: PS-ThP-22, 43
 Nguyen, Chi Thang: AP+AS+EL+EM+PS+TF-ThM-6, 31
 Nguyen, Diep: PS2-WeA-5, 28
 Nguyen, Thi-Thuy-Nga: AP+PS+TF-WeA-16, 25; AP+PS+TF-WeA-4, **22**
 Nolan, Michael: AP+AS+EL+EM+PS+TF-ThM-1, **31**
 Nordan, Zach: PS-ThP-21, 43
 Nuwal, Nakul: PS-MoA-1, 4; PS-MoA-14, **6**; PS-MoA-9, 5; PS-ThP-35, 46
 Nye de Castro, Rachel A.:
 AP+AS+EL+EM+PS+TF-ThM-16, 33
— O —
 Obara, Soken: AP+PS+TF-WeM-13, 17
 O'Dea, Daniel: PS-ThP-21, 43
 Oehrlein, Gottlieb: PS-MoM-11, 2
 Oehrlein, Gottlieb S.: PS-MoM-4, 1
 Oh, Ga Hee: PS-ThP-30, 45
 Oh, Il kwon: AP+PS+TF-WeM-7, 17
 Okamoto, Kazuki: EM1+CPS+MS+PS+SM+TF-TuA-3, 12
 O'Neal, Sabine:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, 7
 Oron, Dan:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-7, 7
 Orrière, Thomas: PS-WeM-5, 19
 Otaka, Yuhei: AP+PS+TF-WeM-14, 18
 Overzet, Lawrence: PS-ThP-40, 47

Author Index

- Owen, James: AP+PS+TF-ThA-6, **38**
Ozdemir, Erdem: EM1+CPS+MS+PS+SM+TF-TuA-3, **12**
Ozel, Taner: PS-TuM-14, **10**; PS-TuM-17, **11**
Ozturk, Huseyin: PS-WeM-17, **21**
— **P** —
Pachchigar, Vivek: PS2-TuA-10, **13**
Paddubrouskaya, Hanna: AP+PS+TF-WeA-3, **22**
Pagan, Darren: PS3+TF-FrM-14, **52**
Pai, David: PS-WeM-5, **19**
Paiste, Jacob: PS+AIML-ThA-5, **39**
Panagiotopoulos, Athanasios:
AP+AS+EL+EM+PS+TF-ThM-5, **31**
Pandey, Sumeet: PS+AIML-ThA-4, **39**
Panneerchelvam, Prem: PS+AIML-ThA-3, **39**;
PS-TuM-13, **10**
Paolillo, Sara: PS-MoM-14, **3**
Papanu, James: PS1-TuA-1, **13**
Paquette, Michelle:
EM1+CPS+MS+PS+SM+TF-TuA-1, **12**
Paranjape, Purva: PS-ThP-35, **46**
Pareek, Pankaj: PS-WeM-5, **19**
Park, Jae Hyung: PS-ThP-15, **42**
Park, Jaeseoung: AP+PS+TF-WeA-6, **23**
Park, Jeong Woo: AP+PS+TF-WeA-6, **23**
Park, Jung Woo: AP+PS+TF-WeM-1, **16**
Park, Min-A: AP+PS+TF-WeA-6, **23**
Park, Seong Yun: PS-ThP-15, **42**
Park, Wongyun: PS1-ThM-1, **34**
Park, Woo Chang: PS-MoM-5, **1**; PS-ThP-31,
45; PS-ThP-8, **41**
Park, Yun Jae: PS-ThP-4, **40**
Parsons, Gregory: AP+AS+EL+EM+PS+TF-ThM-7, **32**; AP+EM+PS+TF-FrM-7, **49**;
AP+PS+TF-WeA-11, **24**; AP+PS+TF-WeM-2,
16
Partridge, Jonathan: AP+PS+TF-WeA-5, **23**
Pastor, Daniel: PS-ThP-33, **45**
Patel, Dhruval: PS3+TF-FrM-12, **51**
Patel, Drhuval: PS-ThP-26, **44**
Pavlidis, Spyridon: EM1+CPS+MS+PS+SM+TF-TuA-4, **12**
Peete, Banks: PS1-ThM-7, **35**
Penny, Christopher: PS-MoM-16, **3**
Perez Lozano, Daniel:
EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, **7**
Perfecto, Eric: PS1-TuA-3, **13**
Perry, Josh: PS-ThP-23, **43**; PS-ThP-24, **43**
Petrova, Lina: PS-ThP-36, **46**
Pezeril, Maxime: PS1-WeA-1, **27**
Phaneuf, Raymond: AP+PS+TF-ThA-1, **37**
Phillippe, Thueille: PS1-WeA-1, **27**
Pickering, Ricky: PS2-ThM-14, **35**
Pickholtz, Sam: PS2-ThM-14, **35**
Picuntureo, Matias: AP+AS+EL+EM+PS+TF-ThM-8, **32**
Picuntureo, Matías: AP+AS+EL+EM+PS+TF-ThM-13, **32**
Pimenta Barros, Patricia: PS1-WeA-1, **27**
Poché, Thomas: PS-MoM-12, **2**
Pokhrel, Ankit:
EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, **7**
Polito, Jordyn: PS1-ThM-3, **34**; PS-MoA-16, **6**
Polprasarn, Kasidapa: PS-WeM-5, **19**
Posseme, Nicolas: PS1-WeA-1, **27**; PS-TuM-2,
8
Prager, James: PS1-ThM-7, **35**; PS-MoA-8, **5**;
PS-ThP-23, **43**; PS-ThP-24, **43**
Pranda, Adam: PS-MoM-11, **2**; PS-MoM-6, **2**
Prestigiacomo, Joseph: AP+PS+TF-WeM-15,
18
— **Q** —
Qerimi, Dren: PS2-ThM-14, **35**; PS2-ThM-15,
36; PS3+TF-FrM-12, **51**; PS-ThP-22, **43**
Qu, Chenhui: PS3-WeA-16, **29**; PS-MoA-3, **4**
Qui, Larry:
EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-8, **8**
Quijada, Manuel: PS-ThP-16, **42**
— **R** —
Raadu, Michael A.: PS3+TF-FrM-11, **51**
Radyjowski, Patryk: AP+PS+TF-ThA-4, **37**
Raitses, Yevgeny: PS1-ThM-6, **34**; PS-ThP-12,
41
Rajavalu, Shiva: PS-ThP-28, **44**
Raley, Angelique: PS1-TuA-1, **13**
Randall, John: AP+PS+TF-ThA-6, **38**
Ranjan, Ravi: AP+AS+EL+EM+PS+TF-ThM-17,
33
Rau, Samantha: AP+PS+TF-WeA-3, **22**
Rauf, Shahid: PS+AIML-ThA-1, **39**; PS3-TuA-
14, **14**; PS3-WeA-12, **29**; PS-MoA-1, **4**; PS-
MoA-14, **6**; PS-MoA-4, **4**; PS-MoA-9, **5**
Rebollar, Jazline: PS2-TuA-10, **13**
Regev, Tom: PS-ThP-34, **45**
Régnier, Marine: AP+PS+TF-WeA-13, **24**
Remington, Eric: PS+AIML-ThA-5, **39**
Rommel, Michael: PS-MoM-4, **1**
Ren, Yutong:
EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-7, **7**
Renaud, Vincent: PS-MoM-14, **3**
Renault, Olivier: PS1-WeA-3, **27**
Rhino, Daniel: PS-MoM-4, **1**
Rival, Arnaud: PS-TuM-2, **8**
Robertson, Jaime: PS2-FrM-5, **50**; PS-ThP-27,
44; PS-ThP-29, **44**
Rodriguez de Marcos, Luis: PS-ThP-16, **42**
Romadanov, Ivan: PS1-ThM-6, **34**
Ronse, Kurt: PS-MoM-13, **3**
Rost, Christina:
EM1+AP+CPS+MS+PS+SM+TF-WeA-2, **26**
Rotondaro, Antonio: AP+PS+TF-WeA-3, **22**
Roy, Indroneil: PS-MoM-10, **2**
Roy, Syamashree: PS-MoM-14, **3**
Rudawski, Nicholas: AP+PS+TF-ThA-2, **37**
Rudolph, Martin: PS3+TF-FrM-11, **51**
Ruel, Simon: PS1-WeA-1, **27**; PS1-WeA-3, **27**
Rushing, James:
EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-3,
7; EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-
8, **8**
Ruzic, David: PS2-FrM-5, **50**; PS2-FrM-8, **51**;
PS2-ThM-14, **35**; PS2-ThM-15, **36**; PS3+TF-
FrM-12, **51**; PS-ThP-21, **43**; PS-ThP-26, **44**;
PS-ThP-28, **44**; PS-ThP-29, **44**
Ruzic, David N: PS-ThP-27, **44**
— **S** —
Sadi, Dihya: PS-WeM-5, **19**
Sahu, Rupali: PS-MoA-1, **4**; PS-MoA-4, **4**; PS-
MoA-9, **5**; PS-ThP-35, **46**
Sakurai, Shunsuke: PS1-FrM-2, **50**
Salden, Antoine: PS2-ThM-16, **36**
Salek, Matt: PS2-ThM-14, **35**
Salem, Bassem: PS1-WeA-1, **27**
Sales, Maria: AP+PS+TF-WeM-17, **19**; PS-
ThP-16, **42**
Sales, Maria G: AP+PS+TF-WeM-15, **18**
Sandoval, Tania: AP+AS+EL+EM+PS+TF-ThM-
8, **32**; AP+EM+PS+TF-FrM-2, **48**
Sandoval, Tania E.: AP+AS+EL+EM+PS+TF-
ThM-13, **32**; AP+AS+EL+EM+PS+TF-ThM-
16, **33**; AP+EM+PS+TF-FrM-1, **48**
Sankaran, Mohan: PS2-TuA-10, **13**
Sankaran, R. Mohan: PS2-WeA-5, **28**
Sarazen, Michele: PS-WeM-3, **19**
Sato, Kiyohiko: PS-MoM-3, **1**
Sato, Noboru: AP+PS+TF-WeM-13, **17**;
AP+PS+TF-WeM-14, **18**
Scheiner, Brett: PS-MoA-3, **4**
Scholl, Wallis: PS-WeM-15, **20**
Schroeder, Manuel: PS-MoA-1, **4**
Schulze, Julian: AP+PS+TF-WeA-10, **24**; PS-
MoA-1, **4**
Scoville, Tim: PS-ThP-34, **45**
Sekine, Makoto: PS-TuM-8, **10**
Semproni, Scott: AP+AS+EL+EM+PS+TF-ThM-
8, **32**
Sengupta, Bratin: AP+AS+EL+EM+PS+TF-
ThM-6, **31**
Seo, Seonghyun: PS1-ThM-1, **34**; PS1-ThM-2,
34; PS1-ThM-5, **34**
Seokhyun, Ha: PS-ThP-32, **45**
Seon Suh, Hyo: PS-MoM-13, **3**
Seong, Inho: PS1-ThM-1, **34**; PS1-ThM-2, **34**;
PS1-ThM-5, **34**
Shah, Miral: PS-ThP-34, **45**
Shannon, Steve: PS1-ThM-7, **35**
Shannon, Steven: PS1-ThM-8, **35**; PS-ThP-25,
43; PS-ThP-34, **45**; PS-WeM-7, **20**
Shannon, Steven C.: PS-TuM-5, **9**
Shapiro, Alex: PS2-FrM-8, **51**
Sharma, Ashish: PS3-WeA-14, **29**
Sharma, Meenakshee: PS-ThP-21, **43**
Shchelkanov, Ivan: PS2-TuA-10, **13**
Shearer, Jeffrey: PS1-TuA-1, **13**
Shekarnoush, Mahsa: PS-ThP-40, **47**
Sherman, Elliot: PS-ThP-21, **43**
Sherpa, Sonam: PS3-WeA-12, **29**
Shi, Xingyi: PS3-WeA-12, **29**; PS-MoA-1, **4**;
PS-MoA-14, **6**
Shi, Zhitian: PS-TuM-6, **9**
Shimogaki, Yukihiro: AP+PS+TF-WeM-13, **17**;
AP+PS+TF-WeM-14, **18**
Shin, Hyeoung: AP+EM+PS+PS+TF-FrM-4, **48**
Shin, Sung Sik: PS-ThP-15, **42**
Shinoda, Kazunori: AP+PS+TF-WeA-16, **25**;
AP+PS+TF-WeA-4, **22**
Shobha, Hosadurga: PS-MoM-16, **3**
Shulze, Julian: PS-MoA-14, **6**
Shvilberg, Liron:
EM1+AP+CPS+MS+PS+SM+TF-WeA-1, **26**;
EM1+AP+CPS+MS+PS+SM+TF-WeA-2, **26**
Simka, Harsono: PS2-ThM-13, **35**
Simmonds, Paul:
EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-3, **7**
Simmonds, Paul J.:
EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-8, **8**
Singh, Harmmeet: PS-TuM-14, **10**; PS-TuM-17,
11; PS-WeM-15, **20**
Singh, Harsimran: PS-MoM-16, **3**
Skidmore, Chloe: EM1+CPS+MS+PS+SM+TF-
TuA-3, **12**
Smith, Carl: PS1-ThM-7, **35**
Smith, Mark: PS-TuM-13, **10**
Smith, Mark D.: PS+AIML-ThA-3, **39**
Smith, Spencer: AP+AS+EL+EM+PS+TF-ThM-
4, **31**
Smith, Taylor G.: AP+PS+TF-WeA-15, **25**
Sohn, Min Kyun: AP+PS+TF-WeA-6, **23**
Solonenko, Dmytro: AP+PS+TF-WeA-10, **24**
Son, Ilseok: PS1-TuA-1, **13**
Son, Sung Hyun: PS1-ThM-6, **34**
Song, Dong Ha: PS-ThP-30, **45**
Song, Hyungseon: PS-ThP-41, **47**
Soulié, Jean-Philippe:
EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, **7**
Souriau, Laurent: PS-MoM-13, **3**; PS-MoM-
14, **3**
Speed, David: PS2-TuA-8, **13**
Sreenilayam, Suveena: AP+PS+TF-ThA-1, **37**
Sriraman, Saravanapriyan: PS3-WeA-16, **29**
Stiegler, Harvey: PS-ThP-40, **47**
Stowell, Michael: PS3+TF-FrM-12, **51**; PS-
ThP-26, **44**

Author Index

Strandwitz, Nicholas:

EM1+CPS+MS+PS+SM+TF-TuA-1, 12
 Suarez Heredero, Lucia: PS-ThP-29, 44
 Suda, Ryutaro: PS-TuM-15, 10
 Suh, Dongwoo: AP+PS+TF-WeA-6, 23
 Suresh Babu, Swetha: PS3+TF-FrM-11, 51
 Surman, Matthew: AP+PS+TF-WeA-5, 23
 Swarup, Jay: AP+EM+PS+TF-FrM-13, 49
 Sydorenko, Dmytro: PS3-WeA-11, 29
— T —
 Tabassum, Nafisa: PS1-ThM-8, **35**
 Tak, Hyun Woo: PS-ThP-2, 40
 Talley, Matt: PS3-WeA-16, 29
 Tamaoki, Naoki: AP+PS+TF-WeM-13, 17;
 AP+PS+TF-WeM-14, 18
 Tan, Samantha: AP+EM+PS+TF-FrM-8, 49
 Taneja, Taareesh: PS3-TuA-14, **14**
 Tarafdar, Raihan: AP+EM+PS+TF-FrM-12, 49
 Tenorio, Jacob A.: AP+AS+EL+EM+PS+TF-
 ThM-4, 31
 Teplyakov, Andrew: AP+EM+PS+TF-FrM-3, **48**
 Tezsevin, Ilker: AP+AS+EL+EM+PS+TF-ThM-8,
 32; AP+EM+PS+TF-FrM-1, 48
 Thapa, Rupak: EM1+CPS+MS+PS+SM+TF-
 TuA-1, 12
 Thelven, Jeremy: AP+EM+PS+TF-FrM-7, **49**
 Thiruppathiraj, Sudharshanaraj: PS3-WeA-14,
 29
 Thomas, Michael: EM1+CPS+MS+PS+SM+TF-
 TuA-4, **12**
 Thyagajaran, Karthik: PS-WeM-5, 19
 Tian, Peng: PS-MoA-1, 4
 Tiwale, Nikhil: PS-TuM-6, 9
 Tochigi, Hidenobu: PS-ThP-14, 42
 Tökei, Zsolt:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-4, 7
 Tomar, Luis: AP+PS+TF-ThA-2, 37; AP+PS+TF-
 WeM-8, 17
 Tomita, Angela: PS2-WeA-5, 28
 Traoré, Aboulaye: AP+PS+TF-WeA-13, 24
 Trendler, Riley: PS-ThP-21, 43
 Trinh, Ngoc Le: AP+PS+TF-WeM-7, 17
 Trolier-McKinstry, Susan:
 EM1+CPS+MS+PS+SM+TF-TuA-2, 12;
 EM1+CPS+MS+PS+SM+TF-TuA-3, 12;
 PS3+TF-FrM-14, 52
 Tsuji, Takashi: PS1-FrM-2, 50
 Tsukune, Atsuhiko: AP+PS+TF-WeM-13, 17;
 AP+PS+TF-WeM-14, 18
 Tsutsumi, Takayoshi: PS-TuM-8, 10
— U —
 Uddi, Mruthunjaya: AP+PS+TF-ThA-4, **37**
 Udovenko, Stanislav: PS3+TF-FrM-14, **52**
 Uhlig, Jan: PS2-ThM-15, 36
 Üner, Necip: PS2-TuA-10, 13
 Uner, Necip Berker: PS-WeM-17, **21**

Upadhyay, Rochan: PS3-WeA-14, 29

— V —
 Vallat, Remi: PS-MoM-14, 3
 Vallat, Rémi: PS-MoM-13, **3**
 van den Biggelaar, Thomas: PS1-ThM-3, 34
 Van Den Heuvel, Dieter: PS-MoM-14, 3
 van der Heijden, Stijn: AP+PS+TF-WeM-5, 17
 van Gurp, Marnix: PS1-ThM-3, 34
 Vauche, Laura: PS1-WeA-1, 27
 Vaufrey, David: PS1-WeA-3, 27
 Vella, Joseph: AP+AS+EL+EM+PS+TF-ThM-3,
31
 Vemuri, Vamseedhara:
 EM1+CPS+MS+PS+SM+TF-TuA-1, 12
 Ventzek, Peter: AP+AS+EL+EM+PS+TF-ThM-
 14, 33; AP+AS+EL+EM+PS+TF-ThM-15, 33;
 PS1-FrM-1, 50; PS-MoA-15, 6
 Verma, Abhishek: PS+AIML-ThA-1, **39**; PS-
 MoA-7, 5
 Verstraete, Lander: PS-MoM-13, 3
 Vong, Man Hou: AP+PS+TF-WeM-2, **16**
 Voronina, Victoria: PS2-FrM-5, 50
 Vu, Karl: PS-ThP-29, 44
— W —
 Wagenaars, Erik: PS-MoA-13, 6
 Walton, Scott: AP+PS+TF-WeM-17, 19; PS3-
 WeA-13, 29; PS-ThP-16, **42**; PS-ThP-36, 46
 Wang, Jun-Chieh: PS-MoA-1, 4
 Wang, Mingmei: AP+PS+TF-ThA-7, **38**; PS3-
 WeA-9, 28; PS-MoA-11, 5; PS-TuM-14, 10;
 PS-TuM-17, 11; PS-WeM-15, 20
 Wang, Tianhong: PS-MoA-1, 4
 Wang, Ting-Ya: AP+AS+EL+EM+PS+TF-ThM-
 14, **33**
 Wang, Xue: PS-TuM-14, 10; PS-TuM-17, 11
 Watkins, Owen: AP+PS+TF-WeA-15, **25**
 Watson, Andrea:
 EM1+AP+CPS+MS+PS+SM+TF-WeA-1, 26
 Way, Doug: PS-WeM-1, 19; PS-WeM-2, 19
 Wellenius, Patrick:
 EM1+CPS+MS+PS+SM+TF-TuA-4, 12
 Wheeler, Virginia: AP+PS+TF-WeM-17, **19**;
 PS3-WeA-13, 29; PS-ThP-16, 42
 Wheeler, Virginia D: AP+PS+TF-WeM-15, **18**
 Wiggers, Hartmut: PS-WeM-13, **20**
 Williams, Ekow:
 EM1+AP+CPS+MS+PS+QS+SM+TF-TuM-8, 8
 Winter, Leonhard: AP+AS+EL+EM+PS+TF-
 ThM-17, **33**
 Wolden, Colin: PS-WeM-1, 19; PS-WeM-2, **19**
 Wong, Eric Ha Kit: AP+EM+PS+TF-FrM-1, **48**
 Woodward, Jeffrey: PS3-WeA-13, 29
 Wubs, Jente: PS1-ThM-3, **34**; PS2-ThM-16,
 36
— X —
 Xia, Longrui: AP+PS+TF-ThA-7, 38
 Xie, Jin: PS-TuM-13, 10

Xie, Xikae: EM1+AP+CPS+MS+PS+QS+SM+TF-
 TuM-8, 8
 Xu, Qing: PS3-WeA-9, 28
— Y —
 Yakunin, Andrei: PS2-FrM-5, 50
 Yamaguchi, Jun: AP+PS+TF-WeM-13, 17;
 AP+PS+TF-WeM-14, 18
 Yamasaki, Yutaka: PS1-TuA-1, 13
 Yan, Dayun: PS-MoM-16, 3
 Yanagawa, Takumi: PS3-WeA-12, 29
 Yang, Heejae: PS-ThP-19, 43
 Yang, Jinyu: PS-ThP-18, **42**
 Yang, Su Jeong: PS-ThP-8, 41
 Yang, Tsung-Hsuan: AP+AS+EL+EM+PS+TF-
 ThM-15, **33**
 Yanguas-Gil, Angel: AP+AS+EL+EM+PS+TF-
 ThM-6, **31**
 Yasmeen, Sumaira: PS2-ThM-13, **35**
 Yee, Benjamin: PS-MoA-3, 4
 Yeom, Geun Yeong: PS-ThP-8, 41
 Yeom, Geun Young: PS-MoM-5, 1; PS-ThP-2,
 40; PS-ThP-30, 45; PS-ThP-31, 45; PS-ThP-4,
 40; PS-ThP-6, 40; PS-ThP-7, 40; PS-ThP-9,
 41; PS-TuM-7, 10
 Yeom, Geunyoung: PS-ThP-11, 41
 Yokogawa, Kenetsu: AP+PS+TF-WeA-16, 25;
 AP+PS+TF-WeA-4, 22
 Yook, Yeon Geun: PS-MoA-12, 5; PS-TuM-16,
11; PS-TuM-5, 9
 Yoon, Kook Hyun: PS-ThP-15, 42
 Yoon, Sung-Young: PS1-ThM-7, 35
 Yoshida, Yusuke: PS-MoM-10, 2; PS-MoM-11,
 2; PS-MoM-6, 2
 You, Hae Sung: PS-ThP-15, 42
 You, Shinjae: PS1-ThM-1, 34; PS1-ThM-2, 34;
 PS1-ThM-5, 34
 Yu, Dong Hun: PS-ThP-15, 42
— Z —
 Zachariou, Anna: AP+PS+TF-ThA-2, 37
 Zaera, Francisco: AP+AS+EL+EM+PS+TF-ThM-
 17, 33
 Zafar, Abdullah: PS1-ThM-8, 35
 Zendri, Elisabetta: AP+PS+TF-ThA-1, 37
 Zewdie, Getasew: AP+EM+PS+TF-FrM-4, 48
 Zhang, Wenyu: PS-WeM-15, 20
 Zhao, Jianping: AP+AS+EL+EM+PS+TF-ThM-
 14, 33; AP+AS+EL+EM+PS+TF-ThM-15, 33;
 PS1-FrM-1, 50; PS-MoA-15, 6
 Zhou, Chuanzhen:
 EM1+AP+CPS+MS+PS+SM+TF-WeA-1, 26
 Zhu, Ji: AP+EM+PS+TF-FrM-8, 49
 Ziemba, Tim: PS1-ThM-7, 35
 Ziemba, Timothy: PS-MoA-8, 5; PS-ThP-23,
 43; PS-ThP-24, **43**
 Zulqarnain, Syed M.: PS-MoA-8, 5