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

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

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

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

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

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

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

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

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

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

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

Variations in the normalized E_0' amplitude are monitored as function of various substrate temperatures (100–300 °C), plasma powers (100–500 W), and H_2 flow fractions (1.25–6.25%) to capture the interplay between oxide removal, metallic species formation, and plasma-induced disorder with greater sensitivity and immediacy than conventional XPS metrics. These variations can be used to rapidly define practical processing windows and enable efficient down-selection within large plasma parameter spaces. The results highlight a generally useful strategy for integrating in situ critical-point tracking into atomic layer processing of III-V materials and suggest straightforward extensions to other compound semiconductor and dielectric growth environments.

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

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

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

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

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

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

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

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

Acknowledgements

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9:30am **AP+EL+MS+PS+TF-ThM-7 Spectroscopic Ellipsometry for Screening Chemical and Microstructural Properties of MoS_2 Thin Films Grown by Plasma Enhanced ALD**, ***Douglas Heine***[1], University of Michigan, Ann Arbor; *Paula Arellano,* University of Michigan; *Ageeth Bol,* University of Michigan, Ann Arbor

The two-dimensional layered semiconductor MoS_2 has attracted significant interest for applications in next-generation electronics, photonics and electrocatalysis due to its favorable electronic and optical properties. Plasma-enhanced atomic layer deposition (PEALD) provides a scalable route for the growth of MoS_2 thin films, offering excellent thickness control, conformality, and relatively low deposition temperatures, which are advantageous for integration into device architectures. However, PEALD grown MoS_2 films often exhibit complex microstructures, consisting of small and misaligned grains. Such structural disorder can adversely affect device performance due to the strong optical and electronic anisotropy of MoS_2[(1)].

In this presentation, we report the development of a model that rapidly estimates electronic and microstructural properties of PEALD-grown MoS_2 thin films from ellipsometry data. The influence of misaligned, out-of-plane oriented grains ("fins") is described using a Bruggeman effective medium approach for intrinsically anisotropic inclusions[(2)], while local structural disorder is incorporated through modest variations in the intrinsic optical constants. The model is applied to a broad set of films grown under different deposition conditions, and the extracted parameters are correlated with independently measured film properties, including composition, crystallinity and roughness. By enabling rapid insight into the film microstructure and electronic properties, this ellipsometry-based analysis facilitates the development of PEALD processes that yield higher quality MoS_2 films.

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

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

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

Electron beam assisted etching of plasma functionalized materials has emerged as a novel approach to processing damage sensitive materials. This method employs synergistic interactions between low energy electrons and reactive neutral species to enable material removal while mitigating ion bombardment induced damage and achieving higher atomic precision. While this seems to be a promising approach, the influence of electron irradiation on surface chemistry, defect formation and bond scission are not well understood. Clarifying these mechanisms is essential to establishing electron beam-assisted atomic layer etching (EB-ALE) as a viable method for nanoscale fabrication. In this work, we investigate the EB-ALE of chlorinated single crystal silicon (c-Si) surfaces prepared using a remote plasma source with Ar/Cl_2 gas mixtures. The study examines the dependence of surface coverage, material modification and etching behavior on key parameters, including neutral exposure, and flood gun parameters like electron energy and dose delivery. ALE relies on self-limiting surface reactions, and one key question is if the EB-ALE process can maintain a constant etch depth per cycle as surface roughness, defect or byproduct accumulation evolve. The surface evolution is monitored in real time using in-situ ellipsometry, and it is further characterized using X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM) to analyze changes in chemical composition and surface roughness respectively. Together, these measurements will provide a framework to better understand the mechanisms governing EB-ALE and ensure predictive control over low-damage atomic scale processing.

Acknowledgements

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

Atomic layer processing, which consists of atomic layer deposition (ALD) and atomic layer etching (ALE), provides the ultimate processing toolbox for dimensional and compositional control of thin films. In this talk, I will first provide a historical perspective of how specifically, *in situ* techniques implemented for atomic layer processes have impacted our understanding of surface physical and chemical mechanisms that impart atomic layer processes their singular deposition and/or etching characteristics. For example, early work using *in situ* techniques such as, quadrupole mass spectrometry (QMS), Fourier transform infrared spectroscopy (FTIR) and quartz crystal microbalance (QCM) laid the foundation for understanding of the popular ALD chemistries of Al_2O_3 and ZnO. Next, I will showcase the use of *in situ* QMS and spectroscopic ellipsometry (SE) to rapidly screen new precursor molecules and deposit materials for a myriad of applications ranging from lasing fibers to passivation contacts for solar cells. The use of tandem *in situ* techniques will be highlighted which allows for unambiguous determination of both chemical and physical aspects of growth mechanisms during atomic layer processes. Last, due to the massive amounts of data obtained during *in situ* monitoring of processes, machine learning (ML) algorithms can now be successfully used to interpret process performance against process parameters, determining end points and establishing process – property causality. In summary, this presentation highlights the evolution of atomic layer diagnostics from foundational surface chemistry to modern, AI-driven process optimization.

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

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

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[1] **JVST Highlighted Talk**

11:45am **AP+EL+MS+PS+TF-ThM-16 Quantifying Interface Formation in InP/Al_2O_3: ALD Films Probed by XPS and MIS C-V**, ***Fabiano Borges,*** University of Campinas (Unicamp) and Federal Institute of Education, Science and Technology of São Paulo (IFSP), Brazil*; Cassio Almeida,* Semiconductor Laboratory (LabSem), Center for Telecommunication Studies (CETUC), Pontifical Catholic University of Rio de Janeiro (PUC-Rio), Brazil*; Ângela Albuquerque,* Brazilian Nanotechnology National Laboratory (LNNano, Brazilian Center for Research in Energy and Materials (CNPEM)), Brazil*; Gustavo Vieira,* Institute for Advanced Studies (IEAv), Department of Aerospace Science and Technology (DCTA), Brazilian Air Force, Brazil*; José Diniz,* University of Campinas (UNICAMP), Brazil

Abstract

Surface passivation of n-type InP was evaluated by X-ray Photoelectron Spectroscopy (XPS) depth-profile analysis after depositing an Al_2O_3 layer [1] with and without plasma treatments (oxidation (O2), nitriding (N2), oxidation followed by nitriding (O2N2), and nitriding followed by oxidation (N2O2)). To compare samples, we use the atomic fractions of Al2p, In3d, P2p, O1s, N1s, and C1s and two metrics [2]: top-film O/Al ratio at the first depth, with values near 1.5 indicating stoichiometric Al_2O_3; (ii) interface depth, the etch time where the sum of In+P≥20%.

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

Electrical C-V corroborates the chemical trends: the O2 sample exhibits a median flat-band voltage Vfb=0.00V (-0.30V for the control) and a donor density of the same order (2×10^{19} vs 1×10^{19} cm^{-3}), indicating a flatter band alignment without doping degradation. Altogether, O2 delivers the most stoichiometric Al_2O_3, a thicker/interfacial oxide and superior C-V metrics, pointing to improved passivation of InP compared with ALD on untreated surfaces; N2 forms a robust nitride that retards sputter breakthrough; and ALD delivers denser, more stoichiometric Al_2O_3 than sputtering.

References

[1] X. Liu et all, “Interface optimization and performance enhancement of InP MOS capacitors with Sm_2O_3/Al_2O_3 gate stacks,” *Semicond. Sci. Technol.*, vol. 36, n. 10, p. 105013, 2021.

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

Acknowledgments

This research is supported financially by IFSP, FINEP, CNPq and UNICAMP. This research used facilities of the Brazilian Nanotechnology National Laboratory (LNNano), part of the Brazilian Centre for Research in Energy and Materials (CNPEM), a private non-profit organization under the supervision of the Brazilian Ministry for Science, Technology, and Innovations (MCTI). The Spectroscopy and Scattering staff is acknowledged for the assistance during the experiment 20220304. The InP substrate weresupplied by LabSem of PUC-Rio. The author thanks the support of the Instituto de Estudos Avançados of the Brazilian Air Force.

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

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

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

The work presented herein takes this analysis approach one step further by employing MM-SE-based scatterometry *in situ* to monitor 3D patterns during atomic-scale processing. We utilized line gratings that are commercially available from Chipmetrics as Area Selective Deposition Test Chips. We positioned these line gratings in conical mount to access cross-polarization information in the off-diagonal MM elements. We verified that focusing probes were not needed during MM-SE data acquisition with our iSE (J.A. Woollam Co.) instrument mounted on the vacuum reactor. We combined these *in situ* MM-SE measurements with *ex situ* atomic force microscopy (AFM) imaging before and after processing. We studied selective etching of SiN_x and SiO_2 versus Si non-etch, as well as selective etching of SiN_x versus SiO_2 non-etch. These studies demonstrate a feasible characterization of etch selectivity in 3D patterns.

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

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