## Plasma and Vapor Deposition Processes
## Room Town & Country B - Session PP3-ThA

## CVD, ALD, and Laser-based Deposition & Microfabrication Technologies

**Moderators: Carles Corbella**, National Institute of Standards and Technology (NIST)/ University of Maryland, College Park, USA, **Frederic Mercier**, CNRS, Grenoble-INP, University Grenoble Alpes, SIMaP laboratory, France

2:00pm **PP3-ThA-3 Unveiling the Potential of Transparent Conductive Materials by Atomic/Molecular Layer Deposition: From Process Synthesis to Functionalization***, Abderrahime Sekkat [abderrahime.sekkat@toulouse-inp.fr],* Univ. Toulouse, CNRS, Toulouse INP, LGC, Toulouse, France., France **INVITED**

From powering renewable energy systems to transforming lighting and data storage technologies, solar cells, electroluminescent displays (ELDs), organic light-emitting diodes (OLEDs), sensors, and printed electronics are driving the next wave of technological innovation. Transparent conductive materials (TCMs) play a key role in enabling and improving the performance of these devices by offering unique advantages for human–device interfaces and information processing. Today, transparent conducting indium tin oxide (ITO) remains the most widely used TCM, thanks to its excellent optical transparency (>90%) and low sheet resistance (<30 Ω/sq)1,2. It currently holds about 55% of the transparent conductive electrode (TCE) market in 20243. However, ITO is brittle, which limits its use in flexible devices, and its dependence on indium, a critical and scarce resource, raises sustainability concerns. To address these limitations, several alternative TCMs are being actively explored, covering inorganic, metallic, and organic material families.In this presentation, I will give an overview of our ongoing work on developing alternative TCMs using different vapor-phase deposition (VPD) methods. I will first focus on the growth of oxide films using atmospheric pressure spatial atomic layer deposition (AP-SALD), an innovative alternative to conventional ALD4. Unlike traditional ALD, AP-SALD relies on the spatial separation of precursors within a 3D manifold head rather than sequential gas injection. This approach enables faster deposition over large areas, making it well suited for scalable manufacturing. I will present some recent results on p-type oxides obtained by this method5–7. I will then show how oxide coatings can be used to improve the stability of transparent electrodes based on silver nanowire networks8,9. Finally, I will discuss the development of conjugated conductive polymers using oxidative VPD, with examples of their integration into real devices10. Overall, this work illustrates a comprehensive approach, from process synthesis to device functionalization, aimed at advancing the next generation of transparent conductive materials.References1. Nanomaterials 14, 2013 (2024).2. APL Mater. 9, 021121 (2021).3. https://www.imarcgroup.com/transparent-conductive-films-market. (Accessed: 24th July 2025)4. Adv. Mater. Technol. 2000657, 1–8 (2020).5. Nat. Commun. 2022 131 13, 1–11 (2022).6. Commun. Mater. 2, 78 (2021).7. J. Mater. Chem. A 9, 15968–15974 (2021).8. Adv. Mater. Technol. 8, 2200563 (2022).9. Adv. Mater. Technol. 8, 2301143 (2023).10. ACS Appl. Polym. Mater. 5, 10205–10216 (2023).* Corresponding author e-mail: abderrahime.sekkat@toulouse-inp.fr

2:40pm **PP3-ThA-5 In-Plasma XPS: a New Metrology Tool for Semiconductor Process Development and Control***, Andrei Kolmakov [andrei.kolmakov@nist.gov],* NIST-Gaithersburg, USA **INVITED**

Modern ambient pressure X-ray photoelectron spectroscopy (AP-XPS), in addition to its real-time sub-monolayer sensitivity, now covers the pressure range typical of standard plasma processing applications, naturally expanding the capabilities of AP-XPS for operando plasma-assisted control. We recently demonstrated that XPS spectra can be successfully collected under plasma conditions, extending the application of XPS to plasma-surface-liquid-vapor interactions [1]. In previous work [2], we highlighted the importance of plasma chamber wall reactions on sample surface chemistry and showed that plasma-XPS can capture plasma chemistry both at the surface and in the gas phase. We recently applied plasma-XPS to industry-relevant and realistic poorly conducting surfaces, where we observed anomalous XPS binding energy shifts due to sample charging during an AC plasma exposure. We propose mechanisms that explain these plasma-induced shifts. Additionally, we noted plasma-induced binding energy shifts and peak splitting when measuring XPS from the plasma gas phase. The latter can be used for local diagnostics of the local plasma environment.

Overall, plasma-XPS metrology is a new emerging tool that offers significant potential for advancing real-time diagnostics of plasma-assisted deposition processes, and immediate mitigation strategies to reduce the damage of wafers, which is a well-known challenge in semiconductor fabrication [3].

References

[1] J.T. Diulus, A.E. Naclerio, J.A. Boscoboinik, A.R. Head, E. Strelcov, P.R. Kidambi, A. Kolmakov, The Journal of Physical Chemistry C, 128 (2024) 7591-7600.

[2] ] J. T. Diulus, A. R. Head, J. A. Boscoboinik, and A. Kolmakov, J. Vac. Sci. Technol. A 43, 040401, (2025)

[3] K.P. Cheung, Plasma charging damage, Springer-Verlag, London, 2000.

3:20pm **PP3-ThA-7 Ultrathin $SiN_X$ Membrane Stability Under Energy Fluxes from Non-Thermal Plasma Discharges Monitored via Nanocalorimetry***, Carles Corbella [carles.corbellaroca@nist.gov],* National Institute of Standards and Technology (NIST)/ University of Maryland, College Park, USA*; Feng Yi, Andrei Kolmakov,* National Institute of Standards and Technology (NIST), USA

Freestanding ultrathin silicon nitride ($SiN_x$) membranes are widely used as an electron, X-ray, and light transparent windows for environmental spectromicroscopy, separation membranes, and in microelectronics, e.g., as in MEMS devices and nanocalorimeters. However, their stability in the plasma environment requires further studies. Here, suspended 100 nm-thick $SiN_x$ membranes have been wafer-scale fabricated on 15 $mm^2$-silicon frames using lithography. A platinum lithographically defined resistive microsensor of 100 nm thickness is deposited on the backside of the membrane, and it is calibrated for thermometry and calorimetry. This energy flux sensor (nanocalorimeter) has been exposed to cold plasmas in a custom-made research reactor equipped with a remote inductively coupled plasma (ICP) discharge source, Langmuir probe, retarding field energy analyzer, and optical emission spectroscopy (OES) channel. Energy fluxes (ions, electrons, energetic neutrals, and photons) from plasma plume are registered via sensor temperature evolution upon variations in the plasma parameters. The power carried by plasma species can be estimated from a simple energy balance model in measurements using sensor temperature variations up to a few hundred Kelvin with time resolution below 40 ms [Diulus et al, J. Vac. Sci. Technol. B 43, 020601 (2025)]. Additionally, the measurement setup allows for decoupling of the heating contributions by ions and VUV/UV-Vis-IR photons. It was found that the lifetime of the sensor is defined by the $SiN_x$ sputtering rate combined with thermally induced mechanical stress. Ultrathin $SiN_x$ membranes appear to be very robust even when immersed in the RF plasma plume region, manifesting low sputtering yield under typical electrically grounded experimental conditions. To investigate the chemical stability of the ultrathin membranes, nanocalorimetry experiments in argon plasma have been followed by preliminary tests using reactive gases such as oxygen and hydrogen.

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