

ALD for Manufacturing

Room Tampa Bay Salons 1-2 - Session AM1-WeA

ALD Manufacturing Equipment and Processes

Moderators: Tero Pilvi, Beneq, Paul Poodt, SparkNano

1:30pm **AM1-WeA-1 Advanced Batch Atomic Layer Deposition Technology for Future 3D Device**, *Kazuhiro Harada*, KOKUSAI ELECTRIC CORPORATION, Japan

INVITED

Semiconductor logic and memory devices are increasingly being structured in three dimensions, leading to a dramatic rise in demand for Batch Thermal Atomic Layer Deposition processes.

In this context, we will discuss the process trends for each type of 3D device (Logic, NAND, DRAM) and the necessity of Batch Thermal ALD technology.

Batch Thermal ALD enables thin film formation on complex 3D devices while maintaining high quality, coverage, and productivity.

Specifically regarding film quality, the extended time available for each ALD step allows for the formation of extremely high-quality films, even in complex three-dimensional structures.

Furthermore, the latest ALD techniques are being deployed in the industry, not only for conformal deposition but also for seamlessly embedding films into complex shapes and for targeting film formation in specific locations.

Gap fill technology requires not only vertical filling but also the challenging horizontal filling without creating seams.

Additionally, applying Area Selective Atomic Layer Deposition technology to silicon dielectric films is essential, particularly around logic Gate-All-Around (GAA) and 3D NAND cells, to simplify complex integration processes, create 3D structures, and enhance device performance.

To achieve these advanced ALD processes, new precursors and process technologies that precisely control termination, bonding states, steric hindrance, and other factors are required.

We look forward to discussing the evolution of our unique Batch Thermal ALD process for 3D devices and exploring the future prospects of the ALD industry with partners from various technical fields.

2:00pm **AM1-WeA-3 On-Demand Precursor Delivery for Atomic Layer Deposition Using Machine Learning-Based Feedforward Control of Piezoelectric Valves**, *Kanta Ishida*, *Hiroshi Nishizato*, *Shota Oda*, Kumamoto University, Japan; *Yugo Nakaya*, HORIBA STEC, Co., Ltd., Japan; *Kinichi Nasu*, *Hiroshi Okajima*, *Takeshi Momose*, Kumamoto University, Japan

We constructed an on-demand precursor/reactant delivery system through precise flow-rate control enabled by piezoelectric valves. With this system, a precursor is flown only during the precursor dosing step, while stopped during the other three steps. To achieve pressure stability and quick switching of gases equivalent to the conventional run/vent system, steep, ideally stepwise, flow rate changes in opening/closing these valves are mandatory rather than slow changes. Therefore, in-house piezoelectric valves, providing fast response and allowing control of opening ratio over time, were developed, and a recipe to control opening ratio over time was then designed using machine learning and control engineering approach, enabling feedforward control of the valves to achieve these operations. It enables a significant reduction in precursor and reactant consumption during ALD.

Wasting precursors across the three steps, except the precursor dosing step, is a critical issue for making ALD processes environmentally sustainable, especially with run/vent delivery systems. The duration of the precursor dosing is typically reported to be only 1–10% of the ALD cycle [1]. It implies that more than 90% of the precursor is discarded to the vacuum pump without contributing to film growth. To address this issue, establishing an on-demand precursor/reactant delivery system is imperative. However, precise flow-rate control has been challenging due to the transient response caused by gas accumulation upstream of the valve during closure, which rushes into the reactor upon opening.

We characterized the piezoelectric valve and identified that hysteresis between the applied voltage and opening ratio, and nonlinear flow responses, are the main factors hindering precise waveform formation, and, thus, challenging precise flow rate control through the following three phases. First, the transfer function from the opening ratio to the flow rate was derived. Second, to compensate for these nonlinearities, we used machine learning to model the valve behavior and design optimal voltage waveforms that overcome hysteresis. Third, we achieved feedforward

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control based on the developed mathematical model and verified the flow rate. The results demonstrated that the proposed method significantly reduced convergence time to the target flow rate compared with conventional step inputs, resulting in a stepwise gas supply profile.

References [1] O. Graniel *et al.*, *ACS Mater. Au* 3, 296 (2023).

Keywords; On-demand delivery, ALD, Piezoelectric valve, Feedforward control, Sustainability

2:15pm **AM1-WeA-4 High-Aspect-Ratio Integrations: A Path to Full Conformality from HfCl_4 and Select Oxidizers**, *Rong Zhao*, *Eric Condo*, *Bryan Hendrix*, Entegris; *Jimmy Huang*, Entegris, Taiwan

Highly conformal films of HfO_2 by Atomic Layer Deposition (ALD) are critical for future nodes of Complementary Field-Effect Transistor (CFET) logic, advanced high-aspect-ratio (HAR) 3D-NAND flash memory, and future integrated ferroelectric devices. While HfCl_4 -based ALD processes offer superior electrical performance compared to amide-based precursors, ozone (O_3) as a sole co-reactant results in poor growth per cycle (GPC) and restricted penetration into HAR architectures. In this work, we present a systematic evaluation of alternate oxidizer strategies to overcome the intrinsic limitations of O_3 -only processing. The application of mixed O_3 (generated from 20% N_2/O_2 feed gases) and blended $\text{O}_3+\text{N}_2\text{O}$ gases demonstrates significant improvements over baseline, yielding higher GPC, enhanced within wafer (WIW) film uniformity, and increased step coverage on full wafers and test structures with 11:1 aspect ratio (AR). Using optimized conditions at 250–350°C, alternate oxidizer processes achieve near-ideal conformality and uniformity, including ~100% step coverage on 11:1 HAR features, validating their suitability for advanced integration. Postannealing up to 1000 °C confirms film stability, showing negligible shrinkage and consistent refractive index. Film analysis by SIMS supports minimal impurity incorporation indicating clean oxidation pathways and robust compositional control.

Furthermore, Density Functional Theory (DFT) simulations were conducted to investigate adsorption interactions of O_3 and various NO_x species on HfO_2 surfaces. Our studies identified the critical reaction pathway, providing a clear explanation for the improved GPC and step coverage observed with the alternate oxidizers.

Our results establish alternate-oxidizer HfCl_4 ALD as a strong candidate for next-generation logic and memory fabrication, offering scalable improvements in uniformity, conformality, and film purity essential for continued vertical device scaling.

2:30pm **AM1-WeA-5 Stability of MoCl_5 in Heated Canisters and During Delivery**, *Berc Kalanyan*, *James Maslar*, National Institute of Standards and Technology (NIST)

Molybdenum pentachloride (MoCl_5) is an industrially important precursor for applications in atomic layer deposition and etching using fluorine-free chemistry. As a low-volatility solid that is susceptible to hydrolysis, the use of MoCl_5 in manufacturing presents significant challenges in mass transport reproducibility and in-situ generation of impurities, including molybdenum oxychlorides, which are also reactive under process conditions. NIST has been developing various optical methods of detecting MoCl_5 and MoO_xCl_y with high sensitivity and time resolution. This paper will describe the use of UV-vis and infrared absorption spectroscopies and non-dispersive gas analyzers to evaluate the stability of MoCl_5 in canisters and during delivery. Static measurements of the canister headspace in the absence of carrier gas are used to distinguish between trace moisture ingress into the delivery system from finite leaks vs entrainment in the carrier gas. Contributions from the latter moisture source are estimated using a cavity ringdown moisture analyzer installed inline with the carrier gas. The effect of MoCl_5 canister temperature, carrier flow rate, idle time, and pulse duration on mass delivery and impurity generation will be presented. Best practices and implications for deposition and etching applications will also be discussed.

2:45pm **AM1-WeA-6 Precursor-Driven Morphology Tuning in ZnO Grown by ALD on 8-Inch Wafers**, *Katherine Guzey*, *Noah Brechmann*, Fraunhofer IMS, Germany; *Thomas Gemming*, *Marcel Schmickler*, *Harish Parala*, Leibniz Institute for Solid State and Materials Research, IFW Dresden, Germany; *Anjana Devi*, Fraunhofer IMS; Leibniz Institute for Solid State and Materials Research, IFW Dresden; Dresden University of Technology, TU Dresden, Germany; *Nils Boysen*, Fraunhofer IMS, Germany

As a wide-bandgap semiconductor, thin conformal ZnO films are extensively used as transparent electrodes or sensing layers, as well as in other optoelectronic and microelectronic applications. The most commonly used precursor for industrial-scale ZnO ALD is diethylzinc (DEZ), which is pyrophoric and has a non-ideal ALD window. Recently,

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Bis(dimethylaminopropyl) zinc(II) ([Zn(DMP)₂]) has been introduced, exhibiting non-pyrophoric behavior, higher thermal stability, and sufficient volatility for ALD at low temperatures. However, this new precursor has been tested only for ALD in small-scale reactors on small substrates.^{1,2}

In this study, we investigated and optimized a thermal ALD process for growing ZnO on 8-inch wafers using [Zn(DMP)₂] and H₂O as precursors, and compared its performance with the established process using DEZ. Ellipsometry showed that high wafer-scale uniformity was achieved across the entire 8-inch wafer, with 1σ nonuniformities below 1 % at a deposition temperature of 200 °C (Fig. 1a,b). The saturation study for [Zn(DMP)₂] (Fig. 1d) confirmed the self-saturating nature of the process for a pulse time of 1.6 s and above, with a more stable growth per cycle of 1.0 Å to 1.1 Å for [Zn(DMP)₂], especially at higher temperatures (Fig. 1c). This suggests that the latter precursor is more suitable for deposition temperatures above 200 °C. GI-XRD measurements revealed a polycrystalline hexagonal crystal structure with a dominating (002) reflection in all patterns. The texture along the c-axis is significantly enhanced for ZnO films deposited from [Zn(DMP)₂] at higher deposition temperatures.

Analysis by Rutherford backscattering spectrometry (RBS) and X-ray photoelectron spectroscopy (XPS) revealed that highly pure films can be obtained with both precursors within a deposition temperature range of 150 °C to 300 °C. Functional properties were subsequently evaluated by patterning the ZnO films into Van-der-Pauw test structures on 8" wafers to evaluate the sheet resistance (Fig. 3a,b), which was comparable for both precursors (~2200 Ω/sq for [Zn(DMP)₂] and ~700 Ω/sq for DEZ). AFM, GD-OES, and TEM (Fig. 3c-f) further confirmed these results.

In summary, the precursor [Zn(DMP)₂] provides a viable alternative for the ALD of ZnO on 8-inch wafers, which we demonstrated for the first time. Compared to the established DEZ, processes using [Zn(DMP)₂] offer a wider ALD window and a higher crystalline texture along the c-axis, which is highly beneficial for electrical and optical applications. This work therefore paves the way for industrial-scale adoption of the [Zn(DMP)₂] precursor and broadens the options for precise parameter control in ALD-grown ZnO.

3:00pm AM1-WeA-7 Novel Method to Quantify High Surface Area Microloading Effects on Film Conformality, Jussi Kinnunen, Kalle Eskelinen, Chipmetrics Oy, Finland; Stefan Polzin, Chipmetrics GmbH, Germany; Feng Gao, Mikko Utriainen, Chipmetrics Oy, Finland

As device integration moves toward three-dimensional architectures, atomic layer deposition (ALD) increasingly operates under conditions where extreme aspect ratios coexist with strongly varying local surface area loads. In industrial environments, such variations are known to cause microloading effects, where competition for reactant supply leads to local precursor depletion and reduced effective partial pressure, impacting film uniformity and process window stability [1]. However, the magnitude and spatial extent of these effects between neighboring structures with vastly different surface areas remain difficult to quantify using blanket-based monitors.

In this work, we experimentally quantify microloading-induced conformality loss by combining high surface area (HSA) and high aspect ratio (LHAR) test structures within the same ALD process run. Experiments were performed in a Beneq TFS 200 reactor using thermal Al₂O₃ ALD. PillarHall® LHAR5 chips were processed either alone or placed in close proximity to a VHAR1 chip containing a 15 × 15 mm² array of 1 μm diameter, 200 μm deep holes, representing a localized HSA sink. To express the observations in terms of effective reactant supply, the LHAR profiles were analyzed using a Python implementation of the DReaM-ALD diffusion–reaction model [2], based on formulation by Ylilammi et al. [3].

Film conformality was quantified using the penetration depth PD50. Without the VHAR1 HSA structure present, PD50 was 185 μm, corresponding to an effective entrance precursor partial pressure pA0 of 355 Pa. When the LHAR5 chip was placed adjacent to the HSA region, PD50 decreased to 136 μm (195 Pa). At a separation of 5 mm, PD50 recovered to 145 μm (225 Pa), indicating a distance-dependent microloading effect.

The results demonstrate that combining VHAR– and LHAR test structures provide a sensitive and quantitative method to probe microloading effects and local precursor partial pressure variations that remain invisible to blanket wafer measurements. This enables early detection and qualification of layout-dependent conformality risks in mixed-pattern environments, supporting a robust process window definition for high-volume manufacturing.

References

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- [2] E. Verkama et al., DReaM-ALD – Diffusion-Reaction Model for Atomic Layer Deposition, (2023), Github, <https://github.com/Aalto-Puurunen/dream-ald>.
- [3] M. Ylilammi et al., Modeling Growth Kinetics of Thin Films Made by Atomic Layer Deposition in Lateral High-Aspect-Ratio Structures, (2018), *J. of Appl. Phys.*, 123: preprint 205301.

3:15pm AM1-WeA-8 A Solution for Fast Roll-to-Roll High Throughput ALD, Kemi Janssen, Kalpana Systems, Netherlands

ALD is currently the state-of-the-art thin film fabrication technology. Semiconductor industry relies on ALD in its manufacturing process [1]. However, the main drawback of the typical temporal ALD is the slow processing times. This has fuelled the creation of batch processing tools, and more importantly spatial ALD (S-ALD). In S-ALD the spatial separation between the co-reactants replaces the time distinction of precursor pulsing, which results in saving processing times [2].

Nevertheless, scaling up of roll-to-roll (R2R) S-ALD to a manufacturing level with high throughput has been challenging so far. Flexible substrates have become essential to next-generation applications including batteries, solar cells and OLEDs. It is also essential for packaging materials and barrier layers [3].

The challenges entailed in making R2R S-ALD equipment compatible with high volume production environments are mostly related to throughput and up-time. To maximize both simultaneously, perfect precursor separation, to avoid contamination, is necessary. Particularly at high web speeds, i.e. high throughput, this can only be achieved with a small gap between deposition head and substrate. This in turn is challenging when using large, highly flexible substrates.

Kalpana Systems has developed equipment that combines an innovative web transport system with a moving deposition head in such a way that the position of the substrate, relative to the deposition head, is controlled continuously and accurately.

In addition to providing compatibility with large scale production environments our 3 dimensional machine configuration provides the possibility for deposition of complex layer stacks in one process and enables the efficient coating of porous and high aspect ratio substrates.

We will show our first results on coatings for flexible PV, OLED and battery electrodes and relation between coating quality and process parameters.

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- [3] T. Hirvikorpi, R. Laine, M. Vaha-Nissi (2014). Barrier properties of plastic films coated with an Al₂O₃ layer by roll-to-roll atomic layer deposition. *Thin Solid Films* 550, 164–169.

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