ALD Fundamentals: Growth and Characterization
Room HB Plant Ballroom - Session AF2-TuM

Powder ALD

Moderators: Arrelaine Dameron, Forge Nano, **Benjamin Greenberg**, Naval Research Laboratory

10:45am **AF2-TuM-12 ALD on Particulate Materials: Applications & Scale-Up*, J. Ruud van Ommen,*** Delft University of Technology, Netherlands **INVITED**

Atomic layer deposition (ALD) has been extensively investigated for a wide range of applications and is already used commercially in the semiconductor industry, which predominantly relies on planar substrates. However, the intrinsic ability of ALD to coat nearly any surface geometry with atomic-scale thickness control makes it highly appealing for the coating of particulate materials (particles) as well. Recently, we published a review paper with a quantitative analysis of 799 articles from this field, published from 1988 to 2023 [1]. The obtained dataset is the basis for abstractions regarding reactor types (specifically for particles), coating materials, reactants, supports, and processing conditions. Furthermore, the dataset enables direct access to specific processing conditions.

In this presentation, I will give some examples of ALD on particulate materials we have worked on in my group over the past years, such as pharmaceuticals, batteries, catalysts, and rubber tires. ALD can be used to give pharmaceutical particles delayed-release properties. Battery materials provided with an ALD-made, ultrathin coating have a longer lifetime. This approach of reducing degeneration can also be applied to catalysts, while the island-growth mode can be used to make catalysts in a very controlled way. Finally, replacing carbon black with nanosilica can improve the performance of rubber tires. Treating the nanosilica surface with molecular layer deposition can make it less polar, and enhance the mixing with the rubber during production.

For many of the above applications, it is important to process large amounts of particles, to be able to scale up. Scale-up of ALD on particles is quite different from that on wafers, due to the much larger surface area involved. Proper scale-up of ALD on particulate materials encompasses several aspects, including efficient use of reactants, consistent product quality, and safe operation. I will discuss these aspects, and show some different ways of achieving a large-scale process.

[1] Piechulla, P. M., Chen, M., Goulas, A., Puurunen, R. L., & van Ommen, J. R., Chem. Mater. 38(1) (2026) 20–86.

11:15am **AF2-TuM-14 Temperature-Variation Atomic Layer Deposition: A Strategy for Tuning Particle Size and Dispersion toward High-Performance Catalysts***, Manh Duc Dang, Dieu Minh Nguyen,* Phenikaa University, Viet Nam*; Sri Sharath Kulkarni,* ***J. Ruud van Ommen****,* Delft University of Technology, Netherlands*; Hao Van Bui,* Phenikaa University, Viet Nam

Precise control over the metal nanoparticle size and dispersion is critical for the synthesis of high-performance catalysts, yet remains challenging due to particle migration and sintering on substrate surfaces. Here, we introduce a temperature-variation atomic layer deposition (TV-ALD) strategy to regulate nucleation and growth of noble metals, demonstrated for the deposition of sub-nanometer Pt on carbon nanopowders by ALD in fluidized bed reactors. By varying the deposition temperature within each ALD cycle, the interplay between the surface chemisorption and the metal species mobility can be manupulated. In particular, the precursor exposure at elevated temperatures ensures efficient chemisorption and nucleation, while the subsequent oxidant exposure at low temperatures suppresses surface diffusion and coalescence of Pt species. This temperature-variation approach enables the formation of highly dispersed Pt species with significantly narrower particle size distributions compared with conventional isothermal ALD processes. Transmission electron microscopy and surface analyses confirm the presence of sub-nanometer Pt clusters with enhanced dispersion across high-surface-area supports. The improved structural control translates directly into enhanced catalytic performance despite a substantially reduced Pt loading. The proposed TV-ALD strategy demonstrates a simple yet powerful process parameter for controlling the nucleation and growth in ALD, providing a scalable pathway toward the synthesis of size-selective noble metal nanoparticles and highly dispersed catalysts.

11:30am **AF2-TuM-15 Achieving Conformality in Fluidized Bed Atomic Layer Deposition on Ultrafine Cohesive Nanopowders***,* ***Austin Cendejas****, Benjamin Greenberg, Kevin Anderson, James Wollmershauser, Boris Feygelson,* Naval Research Laboratory

Nanoparticles with thin, conformal coatings are of significant interest in a growing number of applications including catalysis, battery technologies, and optoelectronics.[1] Fluidized bed atomic layer deposition (FB-ALD) is a promising technique towards scaling up the batch sizes of these coating processes to industrially relevant scales.[2] A significant challenge emerges as substrate particle size decreases and powders become increasingly “sticky”, or cohesive, and persistent agglomeration directly affects the conformality of coatings. In this work we explore several techniques to improve fluidization behavior and ultimately coating conformality of 35 nm diameter Y_2O_3 nanoparticles. Two modes of agitation are explored including vibration and mechanical stirring. Additionally, two coating materials are employed, Al_2O_3 and MgO, providing insight into contributions from precursor molecule size, sticking coefficient, and partial pressure. Each half-cycle of the FB-ALD process is analyzed not only on the reactor scale, but simultaneously on the microscale as persistent porous agglomerates undergo an infiltration-like ALD diffusion-reaction process on the time scale of precursor pulses.[3,4] Quadrupole mass spectrometry is used for in situ diagnostics of reaction progress and both scanning and transmission electron microscopy accompanied by energy dispersive X-ray spectroscopy are used to assess uniformity and conformality of the ALD coatings.

Funding source: NRL base funds, NRC Postdoctoral Fellowship Program

References:

1. Piechulla, P. M.; Chen, M.; Goulas, A.; Puurunen, R. L.; van Ommen, J. R. Atomic Layer Deposition on Particulate Materials from 1988 through 2023: A Quantitative Review of Technologies, Materials, and Applications. Chem. Mater. 2026, 38 (1), 20–86.

2. Yanguas-Gil, A.; Elam, J. W. Modeling Scale-up of Particle Coating by Atomic Layer Deposition. J. Vac. Sci. Technol. A 2024, 43 (1), 012404.

3. Cendejas, A., D. Moher and E. Thimsen (2020). "Modeling atomic layer deposition process parameters to achieve dense nanocrystal-based nanocomposites." Journal of Vacuum Science & Technology A 39(1).

4. Greenberg, B. L., K. P. Anderson, A. G. Jacobs, A. J. Cendejas, J. R. Hajzus, E. A. Patterson, J. A. Wollmershauser and B. N. Feigelson (2023). "Conformal coating of macroscopic nanoparticle compacts with ZnO via atomic layer deposition." Journal of Vacuum Science & Technology A 42(1).

11:45am **AF2-TuM-16 Atomic Layer DepositionEnabled Control of Densification and Grain Size in ZnO Ceramics***,* ***Eric Bissell****, Anna Zachariou, Jacob Furst, Steve Lass,* University of Central Florida*; Nicholas G. Rudawski,* University of Florida, Gainesville*; Fernando Uribe-Romo, Titel Jurca, Kathleen Richardson, Romain Gaume, Parag Banerjee,* University of Central Florida

Atomic layer deposition (ALD) provides a versatile approach for applying conformal, nanometer-scale coatings to ceramic nanopowders, offering a means to influence grain-boundary chemistry during sintering. In prior work, ALD Al_2O_3 coatings on ZnO nanoparticles were shown to suppress grain growth;1 however, when consolidated under hot-pressing conditions, this grain growth suppression was accompanied by incomplete densification. These observations motivate a more systematic investigation of how sintering parameters affect densification behavior in ALD-modified powder systems.

In this study, ZnO nanoparticles with an average diameter of 60 nm were coated with 10 cycles of ALD Al_2O_3, corresponding to a nominal coating thickness of ~1 nm, and consolidated by hot pressing using a design of experiments (DOE) approach. The effects of maximum hold temperature (850–1000 °C), applied pressure (25–50 MPa), and hold time at peak temperature (1–120 min) were examined while maintaining identical heating ramps, cooling, and tooling conditions. Eight ceramic samples were produced, spanning a representative subset of the processing space.

The resulting ceramics exhibit systematic variations in relative density and average grain size, indicating that densification behavior remains sensitive to processing conditions despite strong ALD-induced grain growth suppression. This behavior is particularly relevant for transparent ceramic systems, where achieving high density while maintaining sub-wavelength grain sizes is necessary to reduce birefringence- and grain-boundary-related optical scattering. Taken together, these results provide an empirical basis for relating sintering parameters to final microstructure in ALD-coated ZnO systems and illustrate how DOE-based process exploration can inform the development of ALD-enabled ceramics with tunable density and grain size.

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