ALD on Particulate Materials

Extended summary of review proposed for IFPRI

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Introduction

Atomic layer deposition (ALD) is a gas-phase coating technique in which the substrate to be coated is consecutively exposed to the reactant gases. Typically, two reactants are used, and the sequencing is repeated a number of times. With this approach, ultrathin films can be created or – depending on the surface energy – clusters or islands are formed on the substrate. In this section, we will give a brief introduction to applying ALD to particles. Moreover, we will compare this approach with chemical vapor deposition (simultaneous feeding of the reactant gases) and liquid-phase coating.

Materials

First, we will discuss the materials that can be deposited using ALD. We will do this using the figure below: it indicates for which materials the ALD chemistry (based on two reactants) has been described in literature.

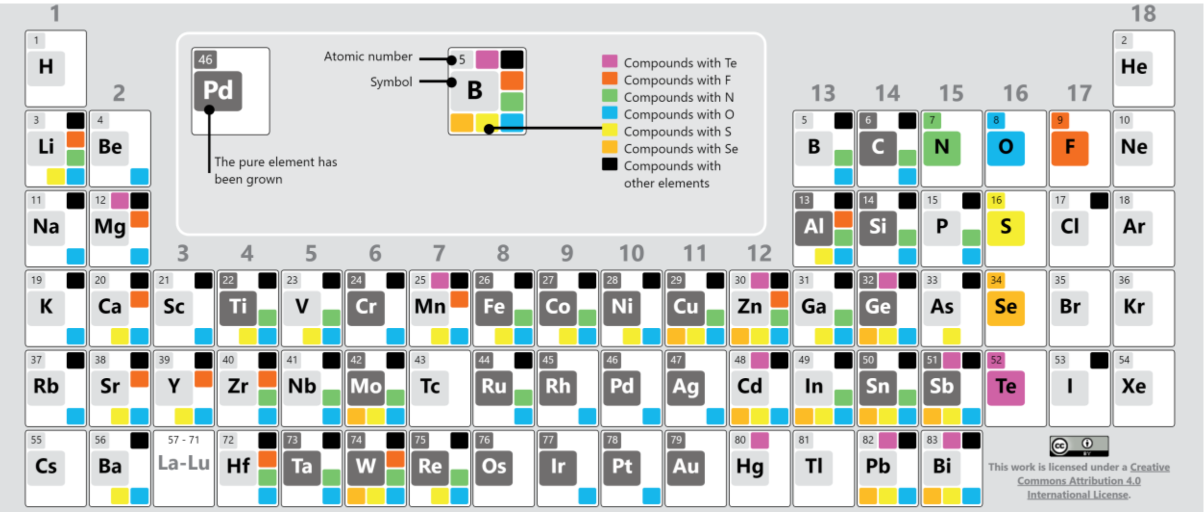

Figure 1. *Overview of the materials grown by ALD. Source: www.atomiclimits.com/alddatabase/, originally based on Miikkulainen, Leskelä, Ritala, Puurunen, J. Appl. Phys. 113 (2013) 021301*

In principle, ALD can be applied to a very broad range of particles. The size of the substrate particles can vary from nm-scale to coarse mm-sized particles. The material of the particles can also vary widely. In literature, most often the coating of metal oxide particles has been described, but also carbon, metal, organic and other materials are reported. To carry out ALD, there should be certain surface groups for "anchoring", e.g. hydroxyl-groups, but if those are not present, they can often be created using an ozone or plasma pretreatment.

Functionalities and applications

In this chapter, we will discuss the various functionalities that can be added to particles using ALD. We list them as the following:

1. ***Coating for protection.*** A very thin coating (often a metal oxide) can protect, for example, cathode particles in batteries against degradation, catalyst particles against poisoning and sintering, and luminescent phosphors in LED lighting against oxidation.
2. ***Coating for controlled released.*** ALD has been shown to provide pharmaceutical powders with an ultrathin coating providing delayed release properties. This approach could also be applied to agro-chemicals such as fertilizers.
3. ***Surface modification to improve flowability & dispersibility***. Particles showing difficult flowing behavior, e.g. because of their organic nature, can be provided with an ultrathin coating (e.g., a metal oxide) to improve the flowability.
4. ***Surface modification to enhance compatibility with matrix***. For particles that poorly mix in a certain matrix (e.g. polar versus apolar), the surface can be modified to obtain a better match with the matrix. We have e.g. demonstrated that mixing silica in rubber works better when first organic groups have been deposited on the silica surface.
5. ***Surface modification to modify visual appearance.*** ALD can be used to change the visual appearance of powders. We have demonstrated that for a standard polyester-based power coating paint, the surface finish can be changed from glow via semi-gloss to matt using ALD of alumina.
6. ***Cluster deposition to create or enhance catalytic activity.*** When the combination of substrate and deposited material is such that island growth is obtained (e.g., deposition noble metal on metal oxide), ALD is an attractive way to make catalysts. Using this approach, the size of the islands (or catalytic clusters) can be very well controlled, with a distribution that is typically much narrower than obtained by liquid-based methods. It can be used to make regular (thermal) catalysts, electrocatalysts and photocatalysts.
7. ***Combinations of 1-6***. The stepwise nature of ALD makes it very well possible to add subsequent treatments: it can be used as a “toolkit”. One can, for example, first deposit catalytic materials, and then provide an ultrathin film to protect it. Another opportunity is that one coating can have multiple functions: the alumina and silica coatings we applied on pharmaceutical particles not only give delayed release properties, but also increase flowability and shelf life.

In the review, we will discuss the different functionalities and application fields in more detail.

ALD processing: from semiconductor applications to particles

Here, we will discuss how process parameters in ALD need to be adapted when moving from coating on wafers for microchips – where it is already widely used – to the processing of particle products. We will focus specifically on particles smaller than 100 µm.

- When processing particles, the surface area is typically orders of magnitude larger. Hence, much larger amounts of reactant will be needed.
- Larger amounts of reactants typically means larger pulse times: often minutes instead of sub-seconds. This has consequences for the processing. It enables modifying conditions such as temperature during one cycle.
- Because of the large amounts of reactants required, it becomes more important to use the reactant as efficiently as possible.
- Although not a perquisite, particle ALD is often carried out at rough vacuum to atmospheric pressure, making the powder handling easier and more cost-effective. ALD on planar substrates is typically carried out at lower pressures.
- When handling particles, the contacting between reactants and particles is an important point. Whether or not mixing the particles is an important issue.

Reactor technology & scale-up

We will discuss the different reactor technologies for applying ALD to particles using Fig. 2.

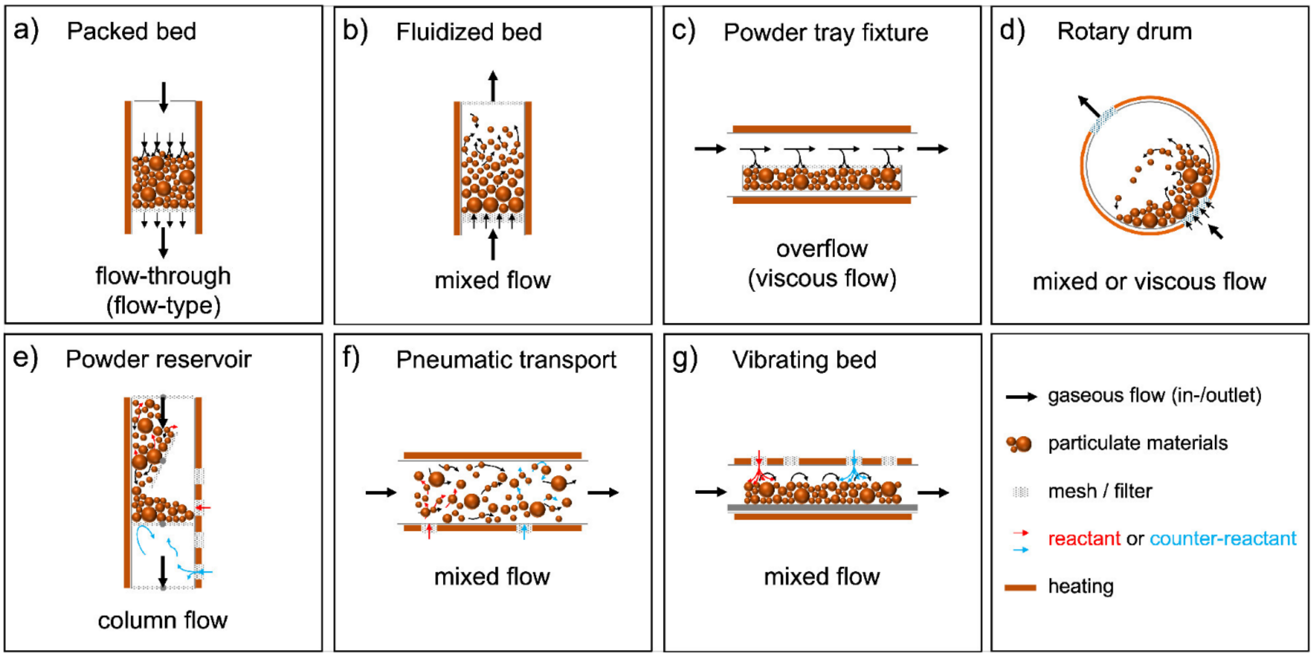

Figure 2. *Operation principle of the four main reactor types (top row): (a) packed bed, (b) fluidized bed, (c) powder tray fixture, and (d) rotary drum. Three spatial ALD concepts (bottom row): (e) powder reservoir, (f) pneumatic transport, and (g) vibrating bed; all*

implemented for manufacturing scale-up. Source: Piechulla, P. M., Chen, M., Goulas, A., Puurunen, R. L., & van Ommen, J. R. (2025). Atomic Layer Deposition on Particulate Materials from 1988 through 2023: A Quantitative Review of Technologies, Materials, and Applications. Chemistry of Materials – in press.

In this figure, panels a-d represent batch-wise operations that are typically used in (academic) research. Panels e-g show continuous processing of ALD on particles: these approaches are better suited for scale up. We will discuss the technical challenges in scaling up. Moreover, a discussion on CAPEX and OPEX (rough estimates) will be included.

Conclusions

We will summarize the main points from the review. In brief, ALD on particulate materials is attractive when ultrathin coatings (typically few nm) are needed. The benefit of ALD is that high precision can be combined with good scalability.

Outlook

We will discuss which new technologies and which new applications can be expected in the coming years.