

Nanostructure Synthesis and Fabrication Room Hall 3 - Session NS-MoP

Nanostructures Synthesis and Fabrication Poster Session

NS-MoP-1 Interfacial Distortion of $\text{Sb}_2\text{Te}_3\text{-Sb}_2\text{Se}_3$ Multilayers via ALD for Enhanced Thermoelectric Properties, Jun Yang, A. Bahrami, S. Lehmann, K. Nielsch, Leibniz IFW-Dresden, Germany

Atomic layer deposition (ALD) is an effective technique for depositing multilayered thin films with precise control of layer thickness and functional properties. In this work, $\text{Sb}_2\text{Te}_3\text{-Sb}_2\text{Se}_3$ multilayers were synthesized using thermal ALD. The presence of both monolayer-by-monolayer and alloy structures when the thicknesses of Sb_2Te_3 and Sb_2Se_3 were reduced to 2 nm. And a decrease in the Sb_2Te_3 layer thickness led to the emergence of distinct peaks from the Laue rings, indicative of a highly textured film structure with optimized crystallinity. Density functional theory simulations revealed that carrier redistribution occurs at the interface to establish charge equilibrium, resulting in band bending. By carefully optimizing the layer thicknesses, we achieved a significant enhancement in the Seebeck coefficient, reaching a peak figure of merit (zT) value of 0.68 at 473 K. These investigations not only provide strong evidence for the potential of ALD manipulation to improve the electrical performance of metal chalcogenide multilayers but also offer valuable insights into achieving high performance in two-dimensional metal chalcogenide systems.

NS-MoP-2 Effect of Ga_2O_3 Doping in Atomic Layer Deposited SnO_2 Thin Films, J. Bae, T. Lee, D. Lee, Hyeon-tag Jeon, Hanyang University, Republic of Korea

Tin dioxide (SnO_2) has suitable characteristics for application to thin film transistor and back-end-of-line (BEOL) devices with low melting point and wide bandgap (3.5~4.0eV). However, as the SnO_2 has property of high conductivity which decreases on/off current ratio, the study for lowering the off current is needed for improving electrical properties. Doping process with atomic layer deposition (ALD) is advantage of reducing off current and controlling precise thickness. Among the dopant elements used as an acceptor in the SnO_2 thin film, Gallium(Ga) has the nearly equal ion radius (0.62Å) to Sn^{4+} (0.69Å). Thus Ga ions can easily replace the position of Sn ions, lowering the crystal lattice mismatch. In addition, from doping process, since doped Ga ions trap the electrons around tin dioxide, off current can be reduced in comparison with none-doped SnO_2 thin film.

In this study, we examined the effect of Ga dopants used as the acceptor in tin oxide with various doping positions of Ga_2O_3 for improving on/off current ratio. Ga doped $\text{SnO}_2(\text{SnO}_2:\text{Ga})$ thin films were deposited using thermal ALD with TDMASn and TMG and ozone. In order to compare the electrical properties with various doping position, Ga doped SnO_2 thin films were deposited at different single Ga_2O_3 doping positions for use as channel layer. The positions of Ga doped layer were controlled by inserting a single Ga_2O_3 cycle into the 31th, 61th, 91th cycles out of 121 cycles(10nm thickness). Auger electron spectroscopy (AES) was used to analyze the atomic concentration of $\text{SnO}_2:\text{Ga}$. It was confirmed that Ga atoms were well located in the site around the doping position. The X-ray photoelectron spectroscopy (XPS) was used for confirming the chemical bonding state of $\text{SnO}_2:\text{Ga}$ thin films. The electrical properties were measured by using semiconductor parameter analyzer (B1500A). The value of on/off current ratio is increased from 5.10×10^3 to 2.78×10^6 compared to none doped SnO_2 . Through this study, we could examine the effect of the Ga doped tin dioxide thin films with various doping position from the comparison of structural, chemical, electrical properties.

NS-MoP-3 Lateral Conversion Synthesis of TMDs from Lithographically Patterned ALD Films, Tevye Kuykendall, Lawrence Berkeley National Laboratory; A. Kemelbay, Lawrence Berkeley National Laboratory, Kazakhstan; S. Aloni, Lawrence Berkeley National Laboratory

Recently, we developed a wafer-scalable method to synthesize lithographically patterned transition metal dichalcogenides (TMDs) using a process that we call "lateral conversion." The method begins with ALD deposited metal-oxide thin-films sandwiched between silica layers. Using photolithography, structures are patterned and etched into the film, exposing the edges of the metal-oxide film. The films are then heated in the presence of chalcogen precursor, which diffuses laterally converting the metal-oxide to TMD. We have demonstrated that TMDs can be synthesized using a variety of metal and chalcogen precursors. Alloys and heterostructures can also be formed by varying the chalcogen precursor

during the conversion process. While the synthesis approach initially relied on photolithography to fabricate structures with micron-scale resolution, we have further reduced the dimensions to achieve sub-20nm features using a block copolymer lithography method that employs the self-assembly of nanoscale polymer structures. We have characterized these TMDs through a variety of techniques including optical microscopy, Raman spectroscopy and High resolution TEM.

NS-MoP-4 Low Temperature SiO_2 Deposition in a Fluidized Bed to Improve the Colloid Stability of Polymer Microspheres, Rens Kamphorst, Delft University of Technology, Netherlands

Fine particles dispersed in a liquid medium have extensive applications in various fields, including food [1], drug delivery [2] and paints [3]. In our study, we investigated the wettability and agglomeration characteristics of polymer microspheres coated with low-temperature deposited SiO_2 (SiCl_4 and H_2O as reactants) in a fluidized bed atomic layer deposition (ALD) setup [4]. Surface characterization by Fourier Transform Infrared Spectrometry (FTIR) revealed the presence of a large number of deposited Si-OH groups within the first cycles, indicating a clear deviation from ideal ALD behavior. Utilizing various characterization techniques, including Environmental Scanning Electron Microscopy (ESEM) and Focused Beam Reflectance Measurement (FBRM), improvements in substrate-water affinity was assessed. Figure 1 displays a clear improvement of substrate-water affinity, even after a single deposition cycle. A drastic decrease in agglomerate size, water contact angle (WCA), and droplet absorption time of the powder was observed when coating was applied. Furthermore, we observed an increase in the amount of Si-OH present on the particle surface with increasing number of cycles, while no significant improvement in water affinity was found after the first coating cycles. Surface coverage was assessed by Low Energy Ion Spectrometry (LEIS), revealing a complete coverage after only 2 deposition cycles. Since no wettability improvements were observed after 2 cycles, we conclude that surface coverage is the primary factor in improving the colloid stability of particles by SiO_2 deposition.

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[3] T.F. Tadros <https://doi.org/10.1002/9783527631179.ch1>

[4] R. Kamphorst et al. <https://doi.org/10.1016/j.surfint.2024.103852>

NS-MoP-7 Atomic Layer Deposition of Ruthenium Nanoparticles on Low Surface Energy Carbon Supports and Titania Nanotube Layers for Alkaline Hydrogen Evolution Reaction, S. Mouli Thalluri, J. Rodriguez-Pereira, R. Zazpe, H. Sopha, University of Pardubice, Czechia; Jan Macak, University of Pardubice, Brno University of Technology, Czechia

Noble metals, such as Pt, Ru, Pd, Ir, etc., have demonstrated superior performance for different catalytic and electrocatalytic applications.^[1] Efforts to reduce or replace these noble metals have been made, due to their scarcity. Atomic Layer Deposition (ALD) is one of the best technique to facilitate lowering of loading mass on a support of interest.^{[2],[3]} Besides, ALD is the most suitable technology for decorating high aspect ratio substrates using noble metal nanoparticles (NPs).^[4] Due to surface energy variations that govern between noble metals and support surfaces, growth initiates as NPs and with further increase in ALD cycles, the agglomeration among NP dominates over the individual NP size increase, thus developing thin films of relatively greater thickness. It is also known that surface energy variations considerably increase the nucleation delay of noble metals, especially for Ru. In this sense, our efforts focused on improving the functionality with pretreatments on carbonaceous supports that showed promise in reducing the nucleation delay of Ru deposited by ALD.

For electrocatalytic applications, it is important to choose the right substrates. Among the available substrates, carbon paper (CP) and titania nanotube (TNT) layers are the best options considering their physiochemical properties, availability, extensive literature and the low costs incurred when using them as support substrates in electrocatalysis and photocatalysis. Various surface modifications for CPs and variations in the morphological aspects of TNT layers have received great attention from applied fields due to their improved surface area, conductivity and stability.^[5-9] The uniform decoration of these CPs and TNT layers by NPs or thin films of catalysts proved to be highly efficient and has no limits in applications.

This presentation will introduce and describe the synthesis of Ruthenium NPs using our ALD tool (Beneq TFS 200) on CP substrates and TNT layers with various aspect ratios. It will also include the corresponding physicochemical and electrochemical characterization and the encouraging results obtained for alkaline hydrogen evolution reaction.

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6. Macak, J. M., Zlamal, M., Krysa, J. & Schmuki, P. *Small* 3, (2007) 300.
7. Liu, C., Sun, C., Gao, Y., Lan, W. & Chen, S. *ACS Omega* 6, (2021) 19153.
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9. Thalluri, S. M. et al. *Ms submitted* (2024)

NS-MoP-8 Iridium Nanoparticles for Alkaline Hydrogen Evolution Reaction Synthesized by Atomic Layer Deposition on Titania Nanotube Layers and Carbon Supports, Jhonatan Rodriguez-Pereira, B. Bawab, R. Zazpe, J. Macak, University of Pardubice, Czechia

The outstanding performance of noble metals such as Pt, Ru, Pd, Ir, etc., for different catalytic applications has been widely demonstrated.^{[1],[2]} However, due to their scarcity, efforts have been made to reduce or substitute these noble metals. Atomic Layer Deposition (ALD) is one of the best technique to facilitate loading mass reduction on a support of interest.^{[3],[4]} Moreover, ALD is the most suitable technology to decorate with noble metal nanoparticles, high aspect ratio and high surface area substrate architectures.^[5] Surface energy variations between the noble metals and the support surfaces cause growth to initiate as nanoparticles (NPs) and as the number of ALD cycles increases, the agglomeration between NPs dominates over the individual NP size increase, thus developing thin films of relatively higher thickness. These surface energy variations considerably increase the nucleation delay of noble metals. In this sense, our efforts focused on improving the functionality with pretreatments on carbonaceous supports that showed promise in reducing the nucleation delay of Ir deposited by ALD.

It is highly important to choose the right substrates for electrocatalytic applications. Among the available substrates, titania nanotube (TNT) layers and carbon paper (CP) are the best options considering their physicochemical properties, availability, extensive literature and the low costs incurred when using them as support substrates in electrocatalysis. Variations in the morphological aspects of TNT layers and several surface modifications for CPs have received great attention from applied fields due to their enhanced surface area, conductivity and stability.^{[6]-[9]} The uniform decoration of these TNT layers and CPs by NPs of catalysts proved to be highly efficient and has virtually no limits in applications.^[10]

The presentation will introduce and describe Iridium NPs synthesis by ALD on TNT layers and CP substrates, including the corresponding physicochemical and electrochemical characterization and the encouraging results obtained for alkaline hydrogen evolution reaction.

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NS-MoP-9 Improving Thermoelectric Performance in Bi-Te Powders through Precision Control of TiO₂-induced Interface via Atomic Layer Deposition, Su Min Eun, B. Choi, Seoul National University of Science and Technology, Republic of Korea

The energy issue and global warming are ever increasing, making it urgent to find green energy conversion technologies. Thermoelectric devices play a crucial role in energy harvesting, as they can directly convert and recycle waste heat into electricity. Significant research is underway to enhance thermoelectric performance such as introducing nanostructures using atomic layer deposition (ALD) above the powder. ALD can control atomic-level film thickness with excellent step coverage and uniformity. ALD thin films increase the interfaces, boosting phonon scattering, and thereby reducing thermal conductivity. Additionally, they induce an energy filtering effect from materials with different band gaps, increasing the Seebeck coefficient from the potential difference. In addition, it can also inhibit grain growth during the sintering process. These approaches enable the

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acquisition of excellent thermoelectric properties, and characteristics of various thermoelectric and thin film materials have been widely reported. Coating TiO₂, known for its high conductivity among oxides and large bandgap, on Bi-Te alloy that demonstrates outstanding thermoelectric performance in the low-temperature range (300-500K) is expected to enhance thermoelectric efficiency. This is anticipated to reduce thermal conductivity while maintaining high electrical properties by phonon scattering and energy filtering effects, overcoming the trade-off relation between them, and thereby improving thermoelectric performance.

In this study, n-type Bi₂Te₃Se_{0.3} (BTS) powders were ground by ball mill for 5hr, and 50, 100, and 200 cycles of TiO₂ layer was coated by ALD with rotary-type reactor using TTIP (Titanium isopropoxide) precursor and H₂O reactant at 200°C. Afterwards, BTS pellet was formed by spark plasma sintering at 50MPa and 673K for 5min. Morphological, structural, and chemical properties representing information such as the shape and distribution of powder or pellet, uniform growth of thin film, and crystallinity were analyzed through X-ray fluorescence, particle size analysis, high-resolution field enhanced scanning electron microscopy, X-ray diffraction, X-ray photoelectron spectroscopy, and Cs-corrected scanning transmission electron microscopy. Thermoelectric properties including thermal conductivity, thermal diffusivity, Seebeck coefficient, and resistance were measured through hall measurement, laser flash analysis, and ZEM-III analysis. Comparing the calculated figure-of-merit (zT) based on measured properties, it was confirmed that interfacial modification through ALD can achieve approximately 27% improvement in thermoelectric performance.

NS-MoP-10 Selective Deposition of Pt Nanoparticles on 2D WS₂ for Enhanced Photodetection, Hwi Yoon, D. Shin, I. Sohn, Yonsei University, Republic of Korea; T. Nakazawa, TANAKA Kikinokogyo K.K, Japan; S. Chung, H. Kim, Yonsei University, Republic of Korea

2D transition metal dichalcogenides (2D TMDCs) with thin and flexible structures have considered promising materials for nanoelectronics technology. In particular, 2D TMDCs-based photodetector is a promising candidate for a visible-light photodetector due to the high mobility and absorption coefficient of TMDC materials. However, the photoresponsivity of 2D TMDCs-based photodetectors is limited by the insufficient presence of photosensitized states caused by inherent surface defects. When exposed to light, the trap sites generated by these defects can capture the photogenerated carriers, leading to a reduction in photosensitivity. Additionally, the presence of unstable defects can introduce impurities such as oxygen and carbon, resulting in a degradation of photoresponsivity.

To enhance the photoresponsivity, it is necessary to develop a method that selectively passivates defects while preserving an active region. Previous research has explored the healing of defects through the adsorption of molecules with thiol groups. However, this approach is difficult to control using solution-based processes and raises concerns about contamination from residue solutions. In contrast, Atomic Layer Deposition (ALD) is a self-limiting process based on surface reaction that allows for precise control of film thickness, nanoparticle size, and coverage at the atomic level. ALD has proven highly effective for selective deposition on chemically reactive defects compared to basal planes. Additionally, the selective deposition of Pt, a representative noble metal, on defects is expected to introduce additional carriers to TMDCs via localized surface plasmon resonance (LSPR). However, there is a lack of research on the selective deposition of nanoparticles (NPs) on 2D TMDCs using ALD Pt.

In this study, we investigated a method for the selective deposition of ALD-based Pt NPs on WS₂, a promising photodetector material, with precise controllability at the sub-nanometer level. For the process development, dimethyl (N,N-dimethyl-3-buten-1-amine-N) platinum (DDAP) (TANAKA, Japan) was utilized as Pt precursors due to reduced steric hindrance resulting from small molecular structure, which allows for precise control of NP size. Specifically, we compared the growth characteristics of Pt NPs on the surfaces of 2D TMDCs based on the type of reactant. We obtained selectively deposited Pt on defects of the WS₂ substrate. Furthermore, we conducted in-depth research on the device performance and its influence based on the size and distribution of Pt NPs. The photocurrent of the Pt NP-functionalized WS₂ photodetectors was significantly higher than that of bare WS₂.

NS-MoP-11 Exploring the Challenges of Atomic Layer Deposition for Graphene Device Manufacturing, Jaspreet Kainth, R. Coleman, R. Baines, H. Glass, Paragraf, UK

Graphene has been speculated to be a suitable material for producing robust and sensitive Hall sensors and biosensors due to its ultra-high carrier mobility. A key challenge however is producing electronic grade, high

mobility graphene at scale. Paragraf is the first company in the world to produce graphene using our proprietary and commercially scalable growth method, directly onto target substrates without the need of a transfer process, using standard semiconductor manufacturing tools.[1] We cover all aspects of production, from the growth of graphene to processing into final devices, resulting in the fabrication of Hall sensors and graphene-FETs.

Graphene is known to be sensitive to its surrounding environments, therefore the impact of the choice of materials, deposition methods and fabrication processes utilised during device manufacture must be well understood with respect to the final device performance. Paragraf employs the use of atomic layer deposition (ALD) as one of its methods for the fabrication of metal oxide thin films, which are required to integrate graphene into devices, since it is well established in producing high-quality films for applications such as high k dielectrics and encapsulation layers to improve device stability. It is well reported that ALD directly onto graphene is a challenge due to its lack of nucleation sites resulting in non-uniform coverage of films or even no film growth, which is a particular challenge for Paragraf due to our extremely low defect level, contaminant free graphene achieved via our particular growth methods. Therefore, development of metal oxide thin film growth via ALD onto graphene has not been trivial. In this work, we will discuss the routes explored to ensure the deposition of high-quality metal oxide films onto graphene, and the challenges with integrating such films into device fabrication processes. By tuning the metal oxide film properties, we have been able to control the final device performance allowing commercially viable devices to be successfully developed that meet our customers' requirements.

Reference:

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NS-MoP-12 Atomic Layer Deposition Assisted Fabrication of Metal-Organic Framework Films for Flexible Biosensing Devices, J. Wang, X. Ke, Z. Zhao, Fudan University, China; X. Zuo, Xueqiang Lu, Jiangsu MNT Micro and Nanotech Co., Ltd., China; G. Huang, Y. Mei, Fudan University, China

Metal-organic framework (MOF) films possess considerable potential application in the field of biosensing devices due to their extensive surface area, abundant active sites, and high catalytic transition metal content. However, the controlled fabrication of MOF films for delicate devices and chips still presents numerous challenges, such as the issues of tight bonding with the substrate, controlled growth on complex substrates, and region-specific fabrication on the substrate. With an induction effect of oxide nanomembrane pre-deposited on substrate by atomic layer deposition (ALD), our group has achieved conformal, dense, uniform, and firmly bonded MOF films on both planar and 3D complex substrates (Figure S1) [1,2]. We then developed patterning techniques for MOF films: patterning the ALD oxide nanomembrane by photolithography enables the precise growth of MOF films with excellent regional selectivity and integration of MOF film into IC chip becomes available (Figure S1) [3,4]. Furthermore, we utilized this ALD assisted technology to prepare MOF film on water-soluble sacrificial layer, and then selectively remove the sacrificial layer, thus leading to preparation of large-area free-standing and transferrable MOF film for advantageous device on fragile substrate (Figure S2) [5]. We have specifically studied the induction effect of the ALD oxide nanomembrane and the growth mechanism of MOF films was disclosed. Several types of MOF films have been prepared by this general strategy, and were engaged in enzyme-free electrochemical biosensing. Due to the uniform and dense nature of the prepared MOF films, flexible biosensor devices towards glucose, lactic acid, dopamine, hydrogen peroxide, etc. with high sensitivities were demonstrated and the devices can be well attached to the human skin (Figure S3) [6-9]. The current ALD assisted approach opens a new window toward the preparation of high-quality MOF films and MOF film-based chips for advantageous sensing applications in post-Moore law period.

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NS-MoP-13 Growth of ALD Gold Nanoparticles on Oxide Surfaces, Mari Napari, H. Cassey, King's College London, UK; A. Werbrouck, University of Missouri; J. Julin, University of Jyväskylä, Finland; S. Barry, Carleton University, Canada; A. Zayats, King's College London, UK

Supported nanoparticles are used in a wide range of applications, including catalysis, photonics, and electronic devices such as sensors. All these applications require precise control of dispersion and size distribution of the nanoparticles for optimal performance, but scalable synthesis of size-selected nanoparticles on large-surface-area supports has proven to be challenging. Atomic layer deposition is often highlighted as a method to produce highly uniform ultra-thin films, but its potential in nanoparticle deposition has been given less attention. However, it has already been shown that ALD can be a viable technique for synthesis of noble-metal nanoparticles, with an option to control the growth by tuning the deposition parameters [1]. There are still remaining challenges in the more widespread use of ALD in nanoparticle synthesis, namely those related to the somewhat poor understanding of the nanoparticle growth and sintering mechanisms during the ALD process, and their dependence on the process parameters and support/substrate material. Here we investigate the use of ALD to grow gold nanoparticles. We use the 3-step ALD Au process developed by Griffiths et al. [2] with trimethylphosphinetrimethylgold, oxygen plasma, and water at 125°C temperature. ALD Au nanoparticles are grown on different oxide surfaces, including SiO₂, Al₂O₃, TiO₂, and alkali-free glass. Particle sizes and size distributions are extracted from SEM and AFM images of samples with 25 - 400 ALD cycles, and the total number of Au atoms per unit area are measured using Rutherford backscattering spectrometry.

To understand the mechanisms of the ALD Au nanoparticle growth the acquired data are analysed using kinetic models, a population-balance model established by Grillo et al. [3], and a novel edge-growth nucleation model. With these models it is possible to distinguish between different nanoparticle growth regimes, where the nucleation of new particles and the evolution of the size distributions are dominated either by hetero/homogeneous reactions of the deposited gold with the substrate and already grown nanoparticles, or by surface diffusion and dynamic coalescence. This information can further be used to design new ALD-based size-selective nanoparticle synthesis processes.

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NS-MoP-14 Multilayered Thin Films for Solid Electrolyte Fabricated by Atomic Layer Deposition, Xiang Yang Kong, A. Fiedler, X. Luo, Shanghai Jiao Tong University, China; S. Indris, Karlsruhe Institute of Technology (KIT), Germany; H. Ehrenberg, Karlsruhe Institute of Technology, Germany

A novel multilayered thin-film solid electrolyte consisting of LiON-AIO_x was synthesized by atomic layer deposition (ALD). The ALD-derived films were grown about 200°C on SiO₂/Si substrate by an alternating deposition of lithium oxynitride (LiON) and AlO_x sublayers with 1–6 nm thickness. The layer-by-layer structure of the films is observed using

cross-sectional HRTEM and confirmed by ToF-SIMS. The ionic conductivities of LiON-AIO_x films are improved by doping with nitrogen in the Li-rich layers. From the electrochemical impedance spectra with elevated temperature, the ionic conductivities of all multilayer LiON-AIO_x films are increased by introducing heterointerfaces with enhanced conductivity along with these heterointerfaces. This is remarkable since the overall concentration of Li⁺ ions is lower in these heterostructures, compared to the single LiON film. An explanation for this behavior could be a high ion mobility at the interfaces, in analogy to layered structures of AlGa_n/Ga_n where a high electron mobility at the interfaces due to the two-dimensional electron gas (2DEG). In this investigation, the increase of ion conductivity could be a result of the two-dimensional ion gas (2DIG) found at the interfaces.

Reference: X. Luo et al., *Solid State Ionics* **373** (2021) 115796

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