

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.

[1] N. Garti et al. <https://doi.org/10.1533/9781845694210.2.149>

[2] N. Singh et al. <https://doi.org/10.1016/B978-0-323-46143-6.00027-0>

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

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

NS-MoP-5 ALD on Particulate Materials: A Comprehensive Review of Processes, Support Materials and Applications, Peter M. Piechulla, M. Chen, Delft University of Technology, Netherlands; R. Puurunen, Aalto University, Finland; J. van Ommen, A. Goulas, Delft University of Technology, Netherlands

Through its course of technology development, atomic layer deposition (ALD) has mostly been applied to flat substrates, typically for applications in the semiconductor industry. Albeit, the inherent processing and coating characteristics of ALD, namely its ability to conformally deposit materials in high aspect ratio geometries with sub-nanometer thickness control, are also attractive for the coating of particulate matter. Application areas requiring substances with large specific surface area, such as heterogeneous catalysis, adsorbents and separations, or highly precise thickness control, such as energy conversion and storage (batteries) or pharmaceuticals are strong drivers for research activities in the realm of ALD on particulate materials. Several scientific review articles on aspects of this field have been published, particularly over the last decade; however, the scope of them is mostly limited to either a processing or an applications point of view.

In this contribution, we present a comprehensive review of the developments in the field of ALD on particles, covering approximately 700 articles in a data-driven rather than anecdotal manner. We target an audience of researchers from the conventional ALD domain who wish to extend their scope of activities to particular media, as well as researchers involved in particle technology who consider ALD as a tool to enhance their technical applications (see supplementary PDF for applications already using ALD on particles). Challenges related to the processibility of powders/particles are addressed by the presentation of an overview of the different reactor engineering approaches implemented. Furthermore, the substrate materials (supports) used are systematically categorized and evaluated with respect to their geometric characteristics (shape and size) but also their surface functionalities. Finally, we cover the range of coating materials attainable and the respective processing conditions; in this way, we also address the similarities and deviations of conventional ALD and ALD on particles in terms of growth characteristics. Through exploring the current state of the art, this review aims to provide both inspiration for new

applications of ALD, and a starting point for researchers in particle-based technologies in search of ALD processes for any given application.

NS-MoP-6 Al₂O₃ and HfO₂ Thin Layers by ALD on 1L-MoS₂: from Nucleation to Structural/Electrical Properties, *Emanuela Schilirò, S. Panasci, B. Galizia, A. Mio, G. Nicotra*, CNR-IMM, Catania, Italy; *S. Agnello*, Department of Physics and Chemistry, University of Palermo; *ATEN Center*, Palermo, Italy; *B. Pecz, G. Radnoczi*, Institute for Technical Physics and Materials Science, Centre for Energy Research, HAS, Hungary; *I. Deretzis, A. La Magna, F. Roccaforte, R. Lo Nigro, F. Giannazzo*, CNR-IMM, Catania, Italy

MoS₂ is one of the most investigated 2D-materials. The great interest to MoS₂ is mainly attributed to its bandgap that, differently from graphene, makes it suitable also for logic and switching devices. However, to guarantee high-performance in terms of field-effect mobility (100-500 cm²/V s), sub-threshold swing (~ 70 mV/dec) and on/off ratio (~ 10⁸) the MoS₂-devices require *high-k* (i.e. Al₂O₃, HfO₂) as top-gate layers. Atomic layer deposition (ALD) is the most appropriate technique to grow uniform high-k layers with accurate thickness control. However, the ALD uniformity on 2D surfaces is typically poor because of the lacking out-of-plane bonds, which should act as nucleation sites. Seed-layers or pre-functionalization are therefore necessary to activate ALD-growth on 2D-materials. On the other hand, their undesired impact on the properties of high-k /2D-materials interface promotes the research of new solutions. In particular, the interaction between 2D-layers and the underlying substrate has been demonstrated to act as driving force of ALD nucleation. In this regard, a monolayer (1L)-MoS₂, exfoliated by gold and maintained on gold-substrate is characterized by nanoscale structural modification (e.g., strain and/or deformation of the MoS₂ layer) which enhances ALD-nucleation providing uniform ultra-thin high-k layers.

In this work, 1L-MoS₂ was exfoliated from molybdenite to a gold substrate. Direct ALD (without seed-layers and pre-functionalization) of Al₂O₃ and HfO₂ were carried-out on MoS₂/Gold substrate, and the structural and insulating properties of high-k were investigated. The ALD coverage was evaluated using standard and conductive-AFM since the early stage of nucleation. During the early deposition cycles, a lower coverage was found for HfO₂ than Al₂O₃, probably due to the higher steric hindrance of the TDMAHF-precursor than TMAI-precursor. However, for longer processes, both high-k films show similar optimal coverage and favorable structural and insulating properties. In particular, uniform and compact layers adherent to 1L-MoS₂ were observed by HR-TEM characterization. Using conductive-AFM, homogeneous insulating behavior has been found for Al₂O₃, while the occurrence of few spots of breakdown events in the current map of HfO₂ is certainly correlated to a lower compactness. Finally, the transfer procedure of high-k/MoS₂ stack from gold to the substrate of interest, crucial for an extensive application of such systems in electronics and optoelectronics, has been optimized.

This work has been supported by the PRIN2022 project “2DIntegratE” and MUR-PNRR project “SAMOTHRACE”.

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 physicochemical 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.

References:

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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.

References:

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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 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 Kikinzoku Kogyo 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 Demonstrating Graphene Hall Sensor Device Stability via Encapsulation Layer Optimization, 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 due to its ultra-high carrier mobility. A key challenge however is producing electronic grade, high mobility graphene at scale. Paragraf has realised this by being 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. One of our products is a magnetic sensor that works based on the Hall effect and has potential applications in current sensing and positioning systems.

The first products were specifically designed for cryogenic and high field applications, where accurate measurements in extremely high fields of 30 T and at cryogenic temperatures are possible simultaneously. Our graphene Hall sensors offer simple, accurate calibration over a wide temperature range, due to high linearity, absence of the planar Hall effect and a repeatable temperature coefficient that is fractions of one percent. This makes them ideal for use in superconductors, quantum computing, and next-generation fusion reactors. The next stage of development is to address larger Hall sensor markets, such as automotive and aerospace. A key requirement for these is continuous use at high temperatures, typically 125 °C and above. Several paths to high temperature stability have been investigated and these are now being developed into commercial devices.

Graphene is known to be sensitive to its surrounding environments, therefore the choice of materials and deposition methods has an impact on the electrical properties of graphene in its final device configuration. We have explored several deposition methods for each layer in the device stack, including atomic layer deposition, chemical vapour deposition, evaporation, and sputtering. Atomic layer deposition is well established for producing high quality encapsulation layers to improve the stability of many device types. In this work, we will discuss how tuning the properties of metal oxide thin films deposited by atomic layer deposition as the encapsulation layer on our graphene Hall sensors has increased our understanding of graphene's electrical performance, allowing commercially viable devices to be successfully developed that meet our customers' requirements and are stable over the lifetime of real-world applications.

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, X. Lu, Jiangsu MNT Micro and Nanotech Co., Ltd., China; G. Huang, Yongfeng 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.

Reference

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Nanostructure Synthesis and Fabrication Room Hall 3E - Session NS-TuA

2D Nanomaterials by ALD (Including Transition Metal Dichalcogenides)

Moderators: Jeffrey W. Elam, Argonne National Laboratory, Chang-Yong Nam, Brookhaven National Laboratory

1:30pm NS-TuA-1 Combining ALD Infiltration and Pressure-Assisted Sintering for Fabrication of Electrically Conductive Nanocomposites, Benjamin Greenberg, K. Anderson, A. Jacobs, A. Cendejas, E. Patterson, J. Wollmershauser, B. Feigelson, U.S. Naval Research Laboratory

Electrically conductive nanocomposites are critical components of electrocatalytic devices, thermoelectric generators, flexible electronics, and many other existing or envisioned technologies. In a disordered conductor/insulator composite with nanoscale domains, high and stable conductivity is achievable provided that (1) the conductive phase does not oxidize or otherwise degrade, (2) the conductive domains percolate—i.e., form continuous networks that traverse the material, and (3) the conductive channels within the network have sufficient cross-sectional area.

In this work, we explore a nascent nanocomposite fabrication strategy based on ALD infiltration and pressure-assisted sintering of nanoparticle compacts. Specifically, we use ALD with long static precursor doses (>10 min) to deposit Al-doped ZnO inside ~2-mm-thick porous compacts of ceramic (e.g., ZnO₂, SiO₂) nanoparticles with ~100 nm diameter, and then we use environmentally controlled pressure-assisted sintering (EC-PAS)¹ at ~2 GPa and 700–1000 °C to remove residual porosity and form fully dense composites. Each ZnO:Al ALD supercycle consists of 15 diethylzinc/water cycles and 1 trimethylaluminum/water cycle, and the deposition temperature is 160 °C except during diethylzinc doses, during which it is lowered to 120 °C to prevent metallic Zn deposition. In principle, this ALD/sintering approach (1) prevents degradation of the ZnO:Al by sealing off the bulk of the composite from the atmosphere, (2) forms percolating ZnO:Al via complete and conformal ALD infiltration, and (3) allows control over the conductive channel cross-sectional area via the number of ALD cycles. In practice, we indeed observe complete infiltration: scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM-EDS) of a representative sample reveals Zn throughout the depth of the composite. Electrical resistivity, however, is surprisingly high: despite deposition of enough ZnO:Al to yield channels with ~7 nm thickness—predicted to enable metallic (band-like) conductivity²—the composite resistivity is >10⁵ Ω-cm. Employing SEM-EDS, x-ray diffraction (XRD), electrical measurements, and other characterization techniques, we investigate potential causes of this high resistivity, which include dopant deactivation and disruption of conductive channels during sintering. We also explore several routes toward achieving metallic conductivity, including *in situ* post-ALD ozone cleaning and optimization of sintering parameters to maintain the doping level and continuity of the ZnO:Al.

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1:45pm NS-TuA-2 Growth of ALD Gold Nanoparticles on Oxide Surfaces, Mari Napari, H. Cossey, 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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2:00pm NS-TuA-3 Tuning MoCl₅ Self-Etching Effect for Deposition of 2D MoS₂ on 300mm Wafer by Thermal ALD, Angelica Azzatli-Zacatzí, N. Vu, D. Lee, T. Ngo, R. Kanjolia, Merck KGaA, Darmstadt, Germany

Crystalline 2D MoS₂ is a semiconductor material with potential application in the next generation of logic and memory devices. The adoption of MoS₂ for future high-volume manufacturing requires the synthesis of high quality and large area 2D material on substrates of interest. The control of MoS₂ film properties such as film uniformity, thickness, and crystallinity are critical for this objective. In the literature, reports of large area deposition of 2D materials include deposition techniques such as chemical vapor deposition (CVD) and atomic layer deposition (ALD). [1] Among the proposed chemical systems for ALD, the MoCl₅-based process has produced crystalline 2D MoS₂ [2,3].

In this work, we investigate the MoCl₅ and H₂S system to deposit MoS₂ by thermal ALD. The effect of the MoCl₅ etch component will be discussed. It was found that self-etching of MoCl₅ can be tuned by process parameters such as precursor pulse time and deposition temperature. The deposition window for 300mm SiO₂/Si wafer substrates will be presented. X-ray fluorescence, Raman spectroscopy, atomic force microscopy, X-ray photoelectron spectroscopy, and transmission electron microscopy were used to characterize the ALD-grown MoS₂. The resulting 2D MoS₂ films exhibit low surface roughness, high uniformity across the wafer, and high level of crystallinity [Fig. 1]. The demonstration of large area and high quality 2D MoS₂ produced by the MoCl₅-based process provides a path toward the implementation of ALD processes for deposition of large area 2D MoS₂.

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- [2] Y. Huang, *et al.*, *Thin Solid Films*, 2017, 624, 101–105.
- [3] Kim, Y., Song, JG., Park, Y. *et al.*, *Scientific Reports*, Vol. 6, 2016, 18754.

2:15pm NS-TuA-4 Low-Temperature ALD of SbO_x/Sb₂Te₃ Multilayers with Boosted Thermoelectric Performance, J. Yang, IFW Dresden, Germany; S. Mukherjee, Jio Institute, India; Sebastian Lehmann, K. Nielsch, IFW Dresden, Germany

Nanoscale superlattice (SL) structures have proven to be effective in enhancing the thermoelectric (TE) properties of thin films. Herein, the main phase of antimony telluride (Sb₂Te₃) thin film with sub-nanometer layers of antimony oxide (SbO_x) is synthesized via atomic layer deposition (ALD) at a low temperature of 80 °C. The SL structure is tailored by varying the cycle numbers of Sb₂Te₃ and SbO_x. A remarkable power factor of 520.8 μW m⁻¹ K⁻² is attained at room temperature when the cycle ratio of SbO_x and Sb₂Te₃ is set at 1:1000 (i.e., SO:ST = 1:1000), corresponding to the highest electrical conductivity of 339.8 S cm⁻¹. The results indicate that at the largest thickness, corresponding to ten ALD cycles, the SbO_x layers act as a potential barrier that filters out the low-energy charge carriers from

contributing to the overall electrical conductivity. In addition to enhancing the scattering of the mid-to-long-wavelength at the $\text{SbO}_x/\text{Sb}_2\text{Te}_3$ interface, the presence of the SbO_x sub-layer induces the confinement effect and strain forces in the Sb_2Te_3 thin film, thereby effectively enhancing the Seebeck coefficient and reducing the thermal conductivity. These findings provide a new perspective on the design of SL-structured TE materials and devices.

2:30pm NS-TuA-5 Enhancing Electrical Properties of 2D WS_2 Grown by ABC PE-ALD with Ion Energy Dose Control, Cindy Lam, E. Kessels, B. Macco, Eindhoven University of Technology, Netherlands

Two-dimensional (2D) transition metal dichalcogenides (TMDCs) are currently on the roadmap of major semiconductor companies as beyond-Si channel material in field-effect transistors (FETs).[1] While 2D TMDCs are typically grown by chemical vapor deposition (CVD), the addition of post-processing steps such as the transfer to specific substrates limits and complicates the overall fabrication process. Accordingly, atomic layer deposition (ALD) is considered a promising method for the direct growth of TMDCs on planar substrates and 3D structures at low temperatures suitable for back-end-of-line (BEOL) processes and its potential for area-selective deposition.[2] However, the mobility of ALD-grown 2D TMDCs substantially falls behind that of CVD-grown 2D TMDCs often due to smaller grain sizes.

In this work, we present that tuning our developed plasma-enhanced ALD (PE-ALD) process which utilizes an inductively coupled Ar plasma (ICP) as an additional C step ($\text{AB} \rightarrow \text{ABC}$) [3],[4] can modify the material properties of WS_2 during film growth. The comparison between WS_2 films grown using the conventional AB and ABC process showcases a significant decrease of the resistivity by four orders of magnitude from $\sim 10^8$ to $\sim 10^4 \mu\Omega\text{-cm}$ due to the improvement of the grain morphology (Fig. 1, SI). While the AB WS_2 film was too resistive for Hall analysis, the ABC WS_2 film exhibited a strong p -type character with a Hall mobility μ_H of $0.78 \text{ cm}^2/\text{Vs}$, which is only one order of magnitude lower than CVD-grown TMDCs. Our results demonstrate effectively improving the electrical properties of WS_2 using the ABC PE-ALD process. Finally, preliminary results on the ion energy $E_{i,\text{flux}}$ and ion flux Γ_i as function of various plasma processing conditions such as the Ar plasma pressure p and plasma exposure time t_p during PE-ALD (Fig. 2 and 3, SI) provide insight into how the material properties can be affected and further tailor them to our specific needs by the use of plasma.

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[3] Balasubramanyam, S. *et al.* *ACS Appl. Mater. and Interfaces*, 12(3), 3873–3885. (2020)

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2:45pm NS-TuA-6 Impact of ALD Precursor Choice on Nucleation and Growth of Dielectrics on 2D Materials, Alexander Shearer, J. Ko, K. Saraswat, E. Pop, S. Bent, Stanford University

2D semiconducting materials have received attention for their potential to facilitate further downscaling of microelectronic devices by overcoming the physical shortcomings of silicon. Due to their inherent atomic thinness and lack of surface dangling bonds, 2D materials can be well-controlled by a gate while preserving the carrier mobility, leading to excellent current switching characteristics, even at the scaling limit. However, the lack of dangling bonds and reactive sites on the surface makes it difficult to deposit a gate oxide by ALD, often leading to sparse nucleation and the growth of films that are low density, defective, rough, and filled with pinholes.

In this work, we employ physisorption-assisted ALD processes and study the nucleation and quality of the deposited dielectrics. We deposit Al_2O_3 on monolayer MoS_2 using a series of ALD precursors in order to investigate the impact of the ALD precursor on nucleation. The precursors utilized in this study are dimethyl aluminum isopropoxide (DMAI), trimethylaluminum (TMA), triethylaluminum (TEA), and a novel Al_2O_3 precursor, triisobutylaluminum (TIBA). Using scanning electron microscopy (SEM), we study film nucleation and continuity as a function of ALD precursor, cycle number, and temperature. The results show that the precursor used in the ALD process dramatically impacts coverage and growth. With the best performing precursor, we observe full coverage of MoS_2 after just 3 nm of material is deposited. After optimizing the temperature and purge time of each process to maximize nucleation, we fabricate transistor devices in which the deposited Al_2O_3 serves as a seed layer for high- k HfO_2 deposition to create the gate stack. Using x-ray photoelectron spectroscopy (XPS) and electrical testing, we investigate the character of the dielectric/ MoS_2 interface. While all three alkyl precursors lead to seed layers and dielectrics

that produce functioning devices, the devices with the best performance are obtained by the precursor that achieves a more uniform, smoother, and denser film, which we attribute to improved nucleation and growth properties. This improved deposition also permits the most thinning of the seed layer. The devices fabricated using the best precursor and improved dielectric show excellent performance such as good on/off ratios (10^6), small device-to-device variation ($\Delta V_T < 1 \text{ V}$), and low effective oxide thickness ($\sim 1 \text{ nm}$). This work provides useful insights into how ALD precursors can be designed to improve the quality of dielectrics on 2D materials, potentially improving the viability of 2D materials for wide ranging applications.

3:00pm NS-TuA-7 Noble Metal Nanoparticles Functionalized 2D Transition Metal Dichalcogenides by Atomic Layer Deposition for Enhanced Sensing Properties Toward Amino Acids, Jisang Yoo, S. Lee, J. Kim, I. Sohn, S. Jung, H. Kim, Yonsei University, Korea

Due to their large surface-to-volume ratio and stability in aqueous environments, two-dimensional (2D) transition metal dichalcogenides (TMDCs) have recently emerged as promising candidates for biosensing materials.¹ Among various biomolecules, amino acids (AAs) have been proposed as one of key biomarkers of human body status and diseases.² The changes in the physiological levels of AAs in biological fluids (e.g., blood, urine) can be used to determine not only nutritional status, but also specific diseases including cancer and diabetes. Therefore, monitoring and detection of AA profile can be of significant importance in potential early diagnostics. Recently, the possibility of detecting AA molecules through direct charge transfer after adsorption on the TMDC surface has been theoretically investigated. Particularly, noble metal (NM) nanoparticles (NPs), such as Au and Pt, functionalization has been proposed to improve the adsorption of biomolecules including AAs, thereby significantly enhancing the sensing properties.^{3,4} However, experimental demonstration of NM NP-functionalized 2D TMDCs for AA sensing is still lacking.

In this study, we aim to develop a biomolecular sensor to detect AAs using Pt NP-functionalized monolayer (ML) WS_2 (Pt- WS_2) channel. Using chemical vapor deposition process, we synthesized grain boundary (GB)-rich ML WS_2 , where GBs can serve as active sites for surface functionalization. By facilitating atomic layer deposition (ALD), Pt NPs were selectively functionalized on GBs of WS_2 . By optimizing the process parameters such as precursor flux, purge time and cycle number, the size and distribution of Pt NPs were controlled precisely. Following the interaction with AA molecules, n -type charge transfer from AA to Pt- WS_2 was confirmed by Raman spectroscopy, photoluminescence and X-ray photoelectron spectroscopy. For measuring the sensing properties, Pt- WS_2 based biosensors were fabricated through lithography process. Enhanced sensitivity and a lower limit of detection compared to pristine WS_2 were observed. Additionally, concentration-dependent linearity curve was established. Finally, enhanced selectivity towards other interfering biomolecules (e.g. dopamine and uric acid) was demonstrated through comparative measurements. This study could be contributed to both basic and expanded research on early disease diagnosis through biomarker monitoring.

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3:15pm NS-TuA-8 Surface Modification of Polyolefin Nonwoven Fabric Through Atomic Layer Deposition (ALD) and Molecular Layer Deposition (MLD), Jae Seok Lee, S. Song, B. Choi, Korea University, Republic of Korea

Polyolefins, renowned for their exceptional mechanical and chemical properties and low density, find extensive applications across diverse fields such as architecture, biomedicine, manufacturing, and environmental sectors. Efforts to supplant conventional metal and ceramic materials with polyolefins are underway by fabricating three-dimensional structural configurations. Nonetheless, the inherent hydrophobicity of polyolefins poses constraints on their applications. Surface modification strategies emerge as viable solutions to address these limitations. While plasma treatment is the conventional approach for surface modification, its propensity to induce polymer damage and its transient effects warrant exploring alternative methodologies.

Surface modification of polyolefin fibers via Atomic Layer Deposition (ALD) and Molecular Layer Deposition (MLD) at low temperatures offers a non-destructive means to achieve targeted surface properties. This method enables the deposition of conformal thin films onto intricate three-

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dimensional structures without compromising the integrity of the fibers. Additionally, the surface modification persists until degradation or delamination of the thin film occurs. Depending on the nature of the deposited thin film, the production of specialized polyolefin fiber materials becomes feasible.

This investigation examines surface property alterations of polyolefin nonwoven fabric by applying Al_2O_3 and polyurea films. Trimethylaluminum (TMA) and H_2O serve as precursors for Al_2O_3 deposition, while 1,4-phenylene diisocyanate (PDIC) and ethylenediamine (ED) are utilized for polyurea formation. The study involves a comparative analysis between intrinsic nonwoven fiber surfaces and those coated with three distinct types of thin films: Al_2O_3 , polyurea, and a layer-by-layer (LBL) configuration employing Al_2O_3 and polyurea layers. The wettability of each fiber is quantified through measurement of the water contact angle (WCA). Furthermore, for polyurea, the adjustability of wettability is explored by varying the terminating groups between isocyanate and amine through alteration of the final precursor. Additionally, the differences in roughness among the fibers, as measured by atomic force microscopy (AFM), provide insights into adjusting surface properties through thin film composition.

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