Monday Afternoon, July 24, 2023

Nanostructure Synthesis and Fabrication Room Regency Ballroom A-C - Session NS-MoA

2D Growth

Moderators: Prof. Dr. John Conley, Oregon State University, Prof. Xiangbo Meng, University of Arkansas

4:00pm NS-MoA-11 A Modified ALD-like Approach to Demonstrate Exceptionally Thin Dielectric Layer Growth on 2D Materials, Daire Cott, S. Sergeant, R. Rennen, G. Benjamin, IMEC Belgium; D. Lin, IMEC, Belgium; X. Wu, IMEC Belgium; Z. Lin, IMEC Belgium,, Belgium; T. Schram, Q. Smets, I. Asselberghs, P. Morin, IMEC Belgium INVITED Uniform deposition of extremely thin dielectrics on 2D materials is sought after to exploit the potential of 2D materials in future nano-electronic devices. To that end, significant challenges must be overcome. The surface of thin layers of pristine 2D materials can be seen as fully passivated where all covalent bonding is located parallel to the plane and layers are bound together by Van der Waals forces. To form an effective interface between the 2D material surface and an oxide covering layer a lowering of the overall surface energy must be achieved. Interestingly, 2D materials already possess an extremely low surface energy (~40 mJ/m²)[1] indicating that wetting is a significant challenge. Additionally, when considering ALD - the method of choice to form scaled high-k dielectric layers on Si device technology - which relies on the ability to form a uniform covalently bound atomic layer as the starting surface to nucleate subsequent layers. Conversely, to maximize 2D materials electronic potential the pristine nature of the 2D material should be maintained without damaging the inplane covalent bonding of the system. This excludes many well-established physical deposition techniques such as PVD sputtering or even plasma enhanced ALD techniques where damage to the 2D laver can be created. inducing defects that although will enhance the nucleation of oxides will simultaneously degrade the electrical performance in nanoscale devices.

In this presentation, a method to overcome the inert nature of 2D materials to support oxide formation without inducing defects into the 2D material will be discussed. Insights into the formation mechanism of thin Aluminum oxide based layers on 2D materials will be outlined with particular focus on two types of transition metal dichalcogenides namely, WS_2 and MOS_2 . Combining our method with 5nm HfO₂ to form the top oxide in lab based dual gated MOSFETs a low equivalent oxide thickness (EOT) of 2.1nm was demonstrated [2], Additionally, the upscaling of this approach to large area 300mm 2D material covered substrates will be also discussed [3]

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[2]: Wu et al., 2021 IEEE International Electron Devices Meeting (IEDM), 7.4. 1-7.4. 4

[3]: Smets et al., 2021 IEEE International Electron Devices Meeting (IEDM). IEDM21-725

4:30pm NS-MoA-13 Water-free SbO_x-ALD-process for Coating Bi₂Te₃particles, Sebastian Lehmann, F. Mitzscherling, S. He, J. Yang, M. Hantusch, A. Bahrami, K. Nielsch, Leibniz Institute for Solid State and Materials Research, Germany

We developed a water-free Atomic Layer Deposition (ALD) process to homogeneously deposit SbOx using SbCl₅ and Sb-Ethoxide as precursors reported for the first time. The coating is applied on Bi2Te3 particles which were synthesized via the solvothermal route to enhance the thermoelectric properties (i.e. Seebeck coefficient, thermal and electrical conductivity) via interface engineering. The amorphous character of the coating was shown by the missing reflexes on the XRD diffractograms. A shift from the oxidation state +III to +V of the Sb-species was observed towards increasing SbOx-coating layer thickness using XPS. Additionally, a peak shift of the Sb $3d_{5/2}$ + O 1s peak indicating increased n-type doping of the material. Electrical measurements of spark plasma sintered bulk samples confirmed the doping effect by a decreased specific resistivity with increasing SbOxlayer thickness. The Seebeck coefficient was improved for the coated samples compared to the uncoated reference sample while the total thermal conductivity remained almost constant resulting in an enhancement of the Power factor and the zT. The results distinctly show that surface engineering via Powder ALD is an effective tool to improve key properties of thermoelectric materials.

4:45pm NS-MoA-14 2D FeS_x Nanosheets by ALD: Electrocatalytic Properties Towards Hydrogen Evolution Reaction, *Raul Zazpe, J. Rodriguez Pereira, S. Thalluri, L. Hromadko,* University of Pardubice, Czechia; *D. Pavliňák, E. Kolíbalová,* Brno University of Technology, Czechia; *H. Sopha, J. Macak,* University of Pardubice, Czechia

In the last years, the search of novel and cost-effective catalysts to replace costly and scarce noble metals drove to explore a wide range of materials, such as transition metal dichalcogenides (TMDCs), phosphides, carbides, nitrides among others.^[1] Inspired by the first experimental evidences reported on the catalytic activity of MoS₂ edge sites,^[2] an intense researching work has been devoted in exploring the catalytic properties of other 2-dimensional (2D) TMDCs, e.g. WS_2 , $MoSe_2$, and $MoTe_2$.^[3-6] 2D materials offer unique structural, optical and electronic properties as compared to the bulk counterparts. In addition, they show significant prospects as catalyst mainly due to the flake-like morphology with minimal thickness, high surface-to-volume ratio, and capability of surface functionalization and tuning the catalytically active edges sites.^[7] Among the TMDCs materials, the catalytic properties of iron sulfide and its different phases, namely FeS2, Fe3S4 and FeS, have been largely overlooked and barely explored compared to other TMDCs, despite the relative low cost and toxicity and the high natural abundance of iron and sulfur.

Herein, we present 2-dimensional (2D) FeS_x nanosheets of different sizes as an electrocatalyst towards hydrogen evolution reaction (HER) in alkaline media. The 2D FeS_x nanosheets were synthesized by applying different numbers of Atomic Layer Deposition (ALD) cycles on TiO_2 nanotube layers and graphite sheets as active supporting materials. The electrochemical results confirmed the electrocatalytic activity with an excellent long-term stability and an enhanced catalytic activity reflected by a noteworthy drop of the HER overpotential. Using a range of characterization techniques, it was unveiled that the origin of the enhanced catalytic activity was caused by the synergistic interplay between in situ morphological and composition changes experienced by the 2D FeS_x nanosheets during HER. Under the application of a cathodic potential in alkaline media, it was observed that the original 2D FeS_x nanosheets transformed to iron oxyhydroxide-iron oxysulfide core-shell nanoparticles, which exhibited a higher active catalytic surface and newly created Fe-based HER catalytic sites. ^[8]

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- [2] T. F. Jaramillo et al Science (80-.).2007, 317, 100–102.
- [3] J. Bonde et al, Faraday Discuss. 2009, 140, 219–231.
- [4] J. C. McGlynn et al., Nat. Commun. 2019, 10, 4916.
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- [7] S. H. Noh et al, J. Mater. Chem. A2018, 6, 20005-20014.

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5:00pm NS-MoA-15 300 mm Wafer-Scale and Self-limiting Layer Synthesis of 2D MoSe₂ by Atomic Layer Deposition, A. Zacatzi, M. Miller, R. Kanjolia, *Thong Ngo*, EMD Electronics

2D Transition metal dichalcogenide (TMD) materials have opened a route to continue the down-scaling trend of semiconductor technology. As part of the TMD materials library, $MoSe_2$ has demonstrated itself to be a very promising candidate as a channel material for transistor devices, achieving field-effect mobilities up to ~ 150 - 200 cm²/(V s). [1]

The synthesis of high quality 2D MoSe₂ is required to unlock the potential application of this material in electronic devices. However, to date, there are limited reports on MoSe2 synthesis in comparison to the variety of techniques used to obtain MoS₂ in the literature. A previous approach describes a two-step MoSe₂ deposition, where MoO₃ is first deposited by atomic layer deposition (ALD) onto a substrate followed by a selenization process performed at 900°C [2]. While this route produced 2D MoSe₂, a single, lower temperature process would be preferred to better meet the high-volume manufacturing demands in the semiconductor industry. In this work, we developed a process to synthesize uniform, 300 mm wafer-scale 2D MoSe₂ films using conventional ALD. A detailed study of the MoSe₂ ALD process window, from 425 °C - 600 °C, correlated with the composition and structural properties of the MoSe₂ film will be presented. The resulting MoSe₂ films from the optimized process are stoichiometric and present a 2H phase. Furthermore, this novel optimized ALD process exhibited a selflimiting layer synthesis-like behavior [3], allowing the precise control over the MoSe₂ thickness.

Monday Afternoon, July 24, 2023

The proposed ALD approach presented in this work contributes to the efforts in developing high-quality 2D materials for the realization of atomically thin channel materials.

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[2] T.-J. Dai, X.-D. Fan, Y.-X. Ren, et al. Mater. Sci. 53, 8436-8444, (2018).

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5:15pm NS-MoA-16 Wafer-Scale Controlled Growth of Two-Dimensional Metal Dichalcogenides Through Atomic Layer Deposition and Top-Bottom Epitaxy, *Chanyoung Yoo*, *W. Choi*, *J. Jeon*, *B. Park*, *G. Jeon*, *S. Jeon*, *C. Hwang*, Department of Materials Science and Engineering and Inter-University Semiconductor Research Center, Seoul National University, Republic of Korea

Two-dimensional (2D) metal dichalcogenides (MDCs) are a class of MX₂ compounds that exhibit unique electrical, optical, and mechanical properties due to their 2D van der Waals (vdW) structures¹. Among them, Sb₂Te₃ is a p-type semiconductor with a narrow direct bandgap that has the potential for applications in a topological insulator², thermoelectric³, and optoelectronic. Sb₂Te₃ is also utilized in phase-change memories, where the pseudobinary GeTe-Sb₂Te₃ tie line compositions are the primary materials⁴. To utilize the unique properties of Sb₂Te₃ and for other 2D MDCs, continuous films with ultrathin thicknesses (<< ~10 nm) must be formed on substrates. However, depositing 2D MDCs such as Sb₂Te₃ with ultrathin thickness is challenging due to their tendency to grow in an island-type non-uniform manner, exhibiting prominent plate-shaped morphologies.

In this study, a novel atomic layer deposition (ALD) method utilizing amorphous (a-) GeTe as a buffer layer was developed to change the islandtype growth of Sb₂Te₃ film to layer-by-layer growth, enabling precise control of film thickness with high uniformity, as shown in Figure 1. The Ge atoms in the buffer layer act as anchoring sites where the Sb-precursors can adhere uniformly. After forming a uniform Sb₂Te₃ layer, the growth was selfterminated by the inert vdW surface (Figure 2a), and the substitutional reaction of Ge atoms with the Sb-precursors dominated the growth. The replacement of Sb atoms formed crystalline (c-) GeSbTe and further Sb₂Te₃ cycles transformed the c-GeSbTe layer into a pure Sb₂Te₃ film (Figure 2b). The self-terminated thickness of the Sb₂Te₃ film was determined by the thickness of the a-GeTe buffer layer (Figure 2c). This new ALD method, referred to as top-to-bottom epitaxial growth, involved depositing the Sb₂Te₃ film through the displacement and diffusion of Sb atoms from the surface to the substrate (see the overall illustrations in Figure 2d). This approach resulted in conformal c-axis-oriented Sb2Te3 films with ideal layerby-layer growth. Furthermore, layer-by-layer growth was maintained on contact hole structures (compare Figure 3a-c and 3d-f), exhibiting c-axis orientation along the curvature without any protruding plates, as evidenced by the characteristic vdW gaps of Sb₂Te₃ parallel to the curved corner. This new ALD method utilizing an amorphous buffer layer holds great potential for application to other 2D MDCs and provides a versatile and scalable platform for their growth.

References

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2	Н.		Zhang	et		al.,		Nat	Phys,	2009.
3	L.	M.	Goncalv	es	et	al.,	Thi	n Solid	Films,	2011.
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Tuesday Afternoon, July 25, 2023

Nanostructure Synthesis and Fabrication Room Regency Ballroom A-C - Session NS-TuA

Nanostructures and Membranes

Moderators: Christian Dussarat, Air Liquide Laboratories, Michelle Paquette, University of Missouri-Kansas City

4:00pm NS-TuA-11 Chemical Vapor Functionalization of Polymer Membranes for Water Treatment, *Jeffrey Elam*, A. Mane, R. Pathak, R. Shevate, V. Rozyyev, Argonne National Laboratory

Polymer membranes are used extensively in water purification to filter and remove particulate and molecular contaminants. Ideally, these membranes should exhibit high permeance, selectivity, and fouling resistance, but these attributes are rarely achieved simultaneously. One approach to improve membrane performance is to modify the polymer using reactive chemical vapors to impart the desired physiochemical properties. In this presentation, I will describe recent work at Argonne using atomic layer deposition (ALD), sequential infiltration synthesis (SIS), and vapor-phase grafting to modify polymer membranes used for ultra- and nano-filtration in water treatment. These techniques rely on self-limiting chemical reactions between gaseous precursors and a solid surface to grow material in an atomically controlled fashion. We have used ALD to produce ultrathin and conformal inorganic layers allowing the membrane pore size and pore wall composition to be precisely tuned, SIS for the bulk modification of polymers by creating an organic-inorganic hybrid material, and vapor-phase grafting of small molecules to achieve additional control over the membrane surface properties. Our studies employ a suite of in-situ and in-operando measurements to elucidate the surface chemistry for these processes and extensive ex-situ characterization and testing to understand the effects of chemical vapor treatment on polymers and how they impact membrane performance.

4:15pm NS-TuA-12 The Molecular Structure of Desalination Polyamides Made by Molecular Layer Deposition, Brian Welch, Technion, Israel; E. Antonio, T. Chaney, O. McIntee, University of Colorado at Boulder; J. Strzalka, Argonne National Laboratory; V. Bright, A. Greenberg, M. Toney, University of Colorado at Boulder; T. Segal-Peretz, Technion, Israel; S. George, University of Colorado at Boulder

The leading method for creating thin, selective polymer films for desalination membranes is interfacial polymerization (IP). Notwithstanding, IP is challenging to control, and isolated films are difficult to develop and study. Molecular layer deposition (MLD), on the other hand, provides a means to synthesize and study polyamide films, and it serves as a promising direct route for membrane fabrication. This work probes the molecular structure of two prominent desalination polyamides synthesized by spatial MLD. Comparisons between the MLD and IP synthesized films shed light on the role of polyamide molecular structure on membrane performance and on the formation of crosslinked polymers via MLD.

MLD with m-phenylenediamine and trimesoyl chloride produced a crosslinked, aromatic polyamide which is prevalent in commercial reverse osmosis membranes, while MLD of piperazine and trimesoyl chloride formed polypiperazine amide, commonly used in nanofiltration membranes. Growth rates were 2.9 and 1.5 Å per cycle at 115°C, respectively. Grazing-incidence x-ray scattering showed oriented molecular spacing features of 3.8 Å in the reverse osmosis polyamide, and larger, isotropic spacing features of 4.6 Å in the nanofiltration films. These orientation and size differences, rather than the crosslink density, correlate to membrane water passage and solute retention.

From ellipsometry and x-ray reflectivity results, we posit that the surface of crosslinked organic MLD films is comprised of polymer tails up to two monomers in length which are connected to a crosslinked bulk layer. These segments are short in comparison to un-crosslinked MLD chemistries whose long segments experience thermal motion which make double reactions more probable with increasing temperature. The structure and surface of MLD and IP polyamide films were compared using x-ray photoelectron spectroscopy, infrared spectroscopy and atomic force microscopy. The MLD films presented properties which are favorable for salt rejection, water flux and fouling resistance: compared to IP, MLD films were dense, highly crosslinked, smooth and homogenous in molecular structure. These results contribute to understanding MLD behavior as well as the synthesis and performance of reverse osmosis and nanofiltration membranes.

4:30pm NS-TuA-13 Deposition of an Atomic Layer Inside Microfluidic Channel, Albert Santoso, J. van Ommen, V. van Steijn, M. David, Y. Hounat, R. Zheng, N. Wijers, J. de Roeck, TU Delft, Netherlands

Since its introduction in the field of microfluidics in 1998, polydimethylsiloxane (PDMS) has seen a rapid increase in its use as a material to construct microfluidic devices. However, its broad application is limited by the absence of its surface anchoring groups and its incompatibility with common organic solvents. Current strategies to address these shortcomings include altering PDMS throughout leading to unwanted changes to the bulk properties or modifying its surface often leading to relatively thick layers.

We present a novel strategy to deposit an atomic layer of metal oxides (TiOx and SiO_x) on the inside of PDMS microfluidic channels using atmospheric pressure atomic layer deposition (AP-ALD). We alternatingly flow nitrogen carrier gas convectively containing the precursors (TDMAT, SiCl₄, ozone, or water) through the microchannels (aspect ratio of 30-200). Infiltration of the porous PDMS walls results in a unique surface-subsurface layer, providing a robust hydrophilic barrier that prevents swelling of PDMS upon contact with organic solvents (Santoso et al. Chemical Communications, 2022). Additionally, little to no changes are observed in the transparency and mechanical property of PDMS. We then studied the use of this coated channel for various applications such as isotope extraction involving common organic solvents. Using the coated chip, we managed to extract 98%±2% natural Ga isotope. As a comparison, the non-coated microfluidic chip leaked and it was difficult to even establish a fluid flow. Using the same set-up, we also deposited TiOx - gold nanoparticle and performed photocatalytic Rhodamine B dye reduction. After 1 minute of contact time, we achieved 30% reduction with comparable kinetic behaviour with previously reported value (Hashemi et.al., Nanoscale, 2020). This study demonstrates ALD as a tool to broaden the application of PDMS devices. This enables the use of PDMS microfluidic system in various high-end applications, positively contributing to the emerging fields involving precision and better contact area such as bioassays and photo-based microreaction.

4:45pm NS-TuA-14 Tunable and Scalable Synthesis of ZnO Nanostructures using ALD Seed Layers, *Alondra M. Ortiz-Ortiz*, *A. Gayle, J. Wang, D. Delgado, D. Penley, H. Faustyn, K. Fuelling,* University of Michigan, Ann Arbor; *A. Bielinski,* Argonne National Laboratory; *C. Sherwood, N. Dasgupta,* University of Michigan, Ann Arbor

There has been significant advancement in the synthesis of nanostructured materials for use in biological, environmental, and energy fields in recent decades, with many novel processes demonstrated at the laboratory scale. Nevertheless, challenges remain at the nanomanufacturing frontier, where it is necessary to maintain tunable material structure and properties using high-volume, high-throughput, and low-cost processes. One of the most facile techniques for the tunable and scalable synthesis of nanostructured materials is atmospheric-pressure hydrothermal synthesis of ZnO, which occurs under mild conditions. We have previously demonstrated that by using Atomic Layer Deposition (ALD) to deposit tunable "seed layers" with sub-monolayer precision in composition and structure, we can programmably tune geometric parameters (spacing, length) of the resulting ZnO nanostructures [1]. We refer to this process as "surface directed assembly", which has been used to enable applications ranging from photocatalysis to anti-biofouling surfaces [2,3].

In this study, we explore a third dimension of programmable control: using ALD seed layers to tune the ZnO nanostructure shape. Specifically, we demonstrate synthesis of Al-Zn-O nanosheets (NSs) using the surface directed assembly process by depositing a seed layer of Al₂O₃. The formation of a zinc-aluminum layered double hydroxide phase occurs from the interactions between the Al and Zn²⁺ ions during the hydrothermal synthesis, as confirmed by X-ray Diffraction (XRD). We demonstrate that the density of the ZnO NSs can be controlled by the number of cycles of Al₂O₃. Furthermore, Scanning Electron Microscopy (SEM) analysis shows that a transition from NS to nanowires (NWs) occurs after extended growth time. To rationalize this behavior, we propose a "phase diagram" for surface directed assembly of nanostructures using ALD. Additionally, we demonstrate the ability to scale-up this surface-directed assembly process onto non-planar, large (cm-scale) surfaces facilitated by the design of a customized flow reactor. This work provides a new pathway to scalable nanomanufacturing enabled by the precise interfacial tunability of ALD for macroscopic applications, including medical devices and marine sensors.

Tuesday Afternoon, July 25, 2023

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5:00pm NS-TuA-15 Block Copolymer Templated HfOx Nanowires – From Fundamental Understanding to Rational Design, *Ruoke Cai*, *T. Segal-peretz*, Technion, Israel

Hafnium oxide (HfO₂) is an attractive material for optoelectronic applications and high- κ dielectrics in semiconductor devices due to its advantageous properties- high dielectric constant, wide band gap, and high stability. However, hafnium oxide nanostructure fabrication currently relies on complex nanofabrication processes. Sequential infiltration synthesis (SIS)- a method derived from atomic layer deposition (ALD), in which vapor phase precursors diffuse into polymers and react with them to form hybrid material, can provide a simple and cost-effective alternative for these processes.

In this study, we demonstrated the formation of hafnium oxide nanostructures- vertical and horizontal nanowires, using block copolymers (BCPs) templates. BCPs were self-assembled into highly ordered and periodic nanostructures, followed by hafnium oxide SIS process. In SIS, selective interactions between the hafnium organometallic precursor and the polar block of the BCP resulted in selective growth within the polar block domains. Following the growth, the BCP template was removed to yield hafnium nanowirestemplated by the BCP morphology. We first explored the precursor-polymer interactions in various homopolymers using in-situquartz crystal microbalance (QCM) microgravimetric measurements. This fundamental understanding was further applied in finding a suitable BCP for templating HfO_2 inorganic nanostructure. We probed the formation of vertical and horizontal nanowires from the BCP templates using high-resolution TEM, EDS, and XPS. This research demonstrates the rational design of the HfO2 nanostructure via template morphology tuning and control over HfO₂ growth.

5:15pm NS-TuA-16 Compressible Polymer Sponge Electrodes via oMLD of PEDOT onto Polyurethane Sponge Supports, *Mahya Mehregan*, *G. Luebbert, K. Brathwaite, Q. Wyatt, E. Throm, D. Stalla, M. Young,* University of Missouri

The formation of compressible porous sponge electrodes is appealing to overcome liquid phase diffusion limitations in applications including electrochemical energy storage, electrochemical water desalination, and electrocatalysis. Previous work has employed wet chemical synthesis to deliver conductive materials into porous polymer sponge supports, but these approaches struggle to produce functional electrodes due to (1) poor electrical connectivity of the conductive network and (2) mechanical rigidity of the foam after coating. In this work we employ oxidative molecular layer deposition (oMLD) via sequential gas-phase exposures of 3,4 ethylenedioxythiophene (EDOT) and molybdenum pentachloride (MoCl₅) oxidant to imbibe polyurethane sponges with electrically-conductive and redox-active poly(3,4 ethylenedioxythiophene) (PEDOT) coatings. We analyze the oMLD deposition on compressive polyurethane sponges and modify the reaction conditions to obtain mechanically compressible and electrically conductive sponges. We specifically identify the importance MoCl₅ dose time to enhance the conductivity of the sponges and the importance of EDOT purge time to preserve the mechanical properties of the sponges. This approach produces an electrically conductive PEDOT network within the sponge support with minimal impact on the sponge's mechanical properties, offering advantages over wet-chemical synthesis approaches. The compressible, conductive sponges we generate have the potential to be used as compressible electrodes for water desalination, energy storage, and electrocatalysis.

5:30pm NS-TuA-17 Low Cost, Large Area Sers Substrates by All Ald Deposited 3d Porous Filter Papers, *Feng Niu*, Raytum Photonics

Surface enhanced Raman scattering (SERS) has emerged as a promising spectroscopic tool for ultrasensitive trace detection of target molecules in the vicinity of nanostructured noble metal surfaces. Researchers have developed many techniques to create SERS substrates with most of them falling into three categories: metal nanoparticles by colloidal chemistry, nano-patterned surfaces, physical and chemical vapor deposition or a combination of them. However, all these techniques have limitations to coating hotspots directly inside 3D SERS substrates. On one hand there is

high demand for super-enhancement, and ultra-sensitivity SERS substrates, on the other hand it is still challenging due to lack of stability, reproducibility, and reusability for such SERS active substrates to enter the realms of real, practical applications. Thus, new concepts, new techniques for simple, larger area, cost-effective fabrication techniques for SERS substrates are still in demand. Atomic layer deposition (ALD) has shown great performance in various fields such as semiconductor development, catalysis, energy and environmental applications. Due to its unique characteristics of super conformity, large area uniformity, easy layer thickness/composition control with precision in atomic scale, low cost and easy scale-up, if designed rationally it is an excellent technique for the bottom-up fabrication of nano-scaled materials and devices. Thus since 2010 ALD has also been investigated for the fabrication of SERS active substrates including Au or Ag nanoparticles (NP), ultra-thin films, conformal coatings of 3D scaffolds, high aspect ratio nanostructures, core-shell nanostructures, tunable nanogaps and high-density hot spots on nanostructured scaffold, etc.

We successfully developed ALD process for making Ag nanoparticles on 3D porous glass fiber filter paper and investigated evolution of Ag NPs with some key process parameters. By fine tuning Ag nanostructures and controlling cycle numbers we were able to obtain high density isolated Ag NPs without formation of agglomerates and continuous Ag films. We demonstrated the presence of strong localized surface plasmon resonance (LSPR) peaks and discovered multiple LSPR peaks associated with multiple scale NP size distributions. We also demonstrated the presence of the SERS signals on these ALD Ag coated glass fiber filter substrates using pyridine as the test analyte. Our results demonstrate that ALD is not only a very promising technique for the rational design as compared to other existing techniques but also has great potential for the fabrication of large area, low-cost nanostructured substrates for commercial applications.

Tuesday Evening, July 25, 2023

Nanostructure Synthesis and Fabrication Room Evergreen Ballroom & Foyer - Session NS-TuP

Nanostructures Synthesis and Fabrication Poster Session

NS-TuP-1 Membrane Property Modification for Energy-efficient Membrane Separations via Vapor Phase Infiltration, Yuri Choe, M. Ong, D. Bergsman, University of Washington

Vapor phase infiltration (VPI) is an emerging method synthesize inorganic materials within polymers using vapor-phase reactants. The incorporation of a secondary molecular species within the polymer via VPI can be used to control the mechanical properties, chemical stability, and thermal resistance of these polymers, making this VPI process applicable to many technologies. For example, VPI has been shown to make commercial membrane filters more stable to organic solvents and high temperatures, potentially enabling their use for emerging separation areas. However, only a handful of VPI process chemistries have been explored. To further expand the properties that can be produced using this technique, more diverse reactants need to be tested. This project focuses on testing organic VPI reactants, such as diethylzinc (DEZ) with ethylene glycol (EG) to synthesize zincones (Zn-organic hybrid material). We expose polyethersulfone (PES) membranes to these reactants, measuring diffusion of reactants and the possible successful reaction of these reactants, along with any changes in the mechanical, thermal, and chemical stability of the resulting hybrid membranes. X-ray photoelectron spectroscopy (XPS) is used to measure any reaction products, while scanning electron microscopy (SEM) along with energy dispersive X-ray (EDX) is used for depth profiling of elemental compositions Ultimately, this project focuses on producing hybrid organicinorganic membranes with greater stabilities at high temperatures and with various chemicals, in the hope that these membranes could be used to separate materials previously inaccessible to polymer membranes, such as organic solvents.

NS-TuP-2 Stacking 2D Chalcogenides Utilizing ALD, D. Shin, J. Yang, F. Krahl, Sebastian Lehmann, K. Nielsch, Leibniz Institute for Solid State and Materials Research, Germany

Chalcogenides, especially transition metal dichalcogenides (TDMCs, but also some other chalcogenides like SnS_2 and Sb_2Se_3) have a layered structure similar to graphene, but instead of being a semimetal they offer a wide variety from semiconducting to conducting materials that are interesting for efficient, fast (and possibly flexible) electronics. Their electronic behavior can be strongly influenced by the thickness of the material (e.g. how many sheets are stacked on top of each other) [1].

With atomic layer deposition (ALD) the layer stacking and individual layer thickness can be precisely controlled in the nm scale and several 2D materials have already been deposited with ALD [2], [3]. ALD is also scalable, unlike other methods for the synthesis of 2D materials e.g. exfoliation.

We utilize ALD to deposit superlattice stacks of 2D materials with "spacing" materials in between to examine their electrical properties. An example is our fabrication of a superlattice consisting of SnS₂ and Sb₂S₃ via ALD but results on other systems including TiS₂, PbS and other sulfides will be presented as well.

References

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NS-TuP-3 Stepwise Growth of Crystalline MoS₂ in Atomic Layer Deposition, A. Cho, S. Ryu, Seong Keun Kim, Korea Institute of Science and Technology, Republic of Korea

Atomic layer deposition (ALD) is considered a promising growth technique for transition metal dichalcogenides (TMDCs) because it ensures uniformity and homogeneity of the TMDC grains. However, the poor crystallinity of

ALD-grown TMDCs remains a critical challenge. Although crystallinity depends on the growth mechanism, the growth behavior of crystalline TMDCs in ALD is unclear. We investigated the growth behavior of highly crystallized molybdenum disulfide (MoS₂) by ALD at 650 C with an extra pulse of remote H₂ plasma. Growth at high temperatures using the activated species aided surface diffusion of the adsorbates. The ALD process facilitates repeated growth and saturation of MoS₂, unlike the normal ALD of 3D bulk materials, where the film thickness monotonically increases with the number of ALD cycles. This unique behavior resulted from the evolution of the basal plane without dangling bonds. On the basal plane. MoS_2 lateral growth dominates vertical growth, and prolonged incubation is required for nucleation on the basal plane. The grain size is small (up to two monolayers) because of the limited mobility on SiO₂, and the grains of the third layer grow to a few hundred nanometers. These findings provide insights into the development of ALD technology for application high-quality to TMDCs.

NS-TuP-4 Electrical Properties of ZnO Nanostructures Derived from Sequential Infiltration Synthesis in Self-Assembled Block Copolymer Patterns: Effects of Alumina Priming, *Won-II Lee*, A. Subramanian, Stony Brook University/Brookhaven National Laboratory; N. Tiwale, K. Kisslinger, Brookhaven National Laboratory; C. Nam, Brookhaven National Laboratory and State University of New York at Stony Brook

Self-assembled block copolymers (BCPs) are promising for the bottom-up, low-cost lithography of functional nanoarchitectures. Especially, BCP thin films can be directly converted into inorganic replicas by sequential infiltration synthesis (SIS), an organic-inorganic hybridization method derived from atomic layer deposition (ALD), which can selectively infiltrate target inorganic materials into one of the polymer blocks in vapor phase. For the high-fidelity infiltration of target materials, alumina is often first infiltrated ("alumina priming") to overcome weak binding of the precursors of target inorganic materials with BCP templates. However, the effects of priming alumina-an electrical insulator-on the electrical properties of the final inorganic nanostructures have been rarely studied. In this work, we investigate the effects of alumina priming on the structural and electrical properties of ZnO nanowire fingerprint patterns fabricated by SIS using diethylzinc (DEZ) and water vapor on the lamellar pattern of self-assembled poly(styrene-b-methyl methacrylate) (PS-b-PMMA) BCP thin film as a function of the amount of infiltrated AlOx contents controlled by trimethylaluminum (TMA) exposure time during a single alumina priming cycle. We find that the characteristic dimension, chemical composition, and electrical conductivity of synthesized ZnO nanostructures could be finetuned by controlling TMA exposure duration. Specifically, increasing TMA exposure time naturally led to improved ZnO infiltration/structural fidelity and increasing feature dimensions (width and height of nanowires), accompanied by elevating Al contents. Counterintuitively, the electrical resistivity of ZnO nanostructure, extracted via transmission line method (TLM) from the two-terminal current-voltage characteristics, was initially decreasing with increasing TMA exposure time, down to 14.3 k Ω ·cm, with corresponding Al concentration of 5.3 at.%., while a further increased TMA exposure duration beyond rendered the resistivity up to two orders of magnitude higher. The observed enhancement in ZnO electrical conductivity by alumina priming could be understood from the well-known case of Al-doped ZnO (AZO), where optimal Al doping in a similar concentration range as in the current study maximizes the ZnO conductivity. The results show that the alumina priming condition typically used for SIS in the field would generally improve the conductivity of infiltration-synthesized ZnO nanostructures, along with their infiltration and structural fidelities.

NS-TuP-5 *in-Situ* XPS Analysis for Wo₃ Sulfurization Process, C. Chang, B. Liu, Taiwan Instrument Research Institute, NARIabs, Taiwan; Yang-Yu Jhang, Taiwan Instrument Research Institute, NARLabs, Taiwan

TMDs are layered materials that can exhibit semiconducting, metallic and even superconducting behavior. In the bulk formula, the semiconducting phases have an indirect band gap. Recently, these layered systems have attracted a great deal of attention mainly due to their complementary electronic properties when compared to other 2D materials. However, these bulk properties could be significantly modified when the system becomes monolayer; the indirect band gap becomes direct. Such changes in the band structure when reducing the thickness have important implications for the development of novel applications, such as high photoluminescence (PL) quantum yield, excellent flexibility, and thermal stability.

Tuesday Evening, July 25, 2023

Previous studies have demonstrated direct sulfurization of the metal precursor as an effective route to produce large-area TMDs. In this paper, we have produced WS₂/SiO₂ by depositing WO_x thin films directly onto Si wafer followed by sulfurization to produce WS₂/SiO₂ heterostructures. However, ALD technique is well known for its thickness controllability, reproducibility, wafer-level thickness uniformity and high conformality. Here, we grew WO_x films by ALD method, and the synthesized WS_2 layer retained the inherent benefits of the ALD process. The overall experiments and measurement were carried out on our homemade 6" cluster systems, which include ALD, RTP, and XPS modules. The sample transfer inside were under 5x10⁻⁶ torr to avoid air pollution. WO_x films were deposited on Si wafer at ALD moduleat ~230°C. After that, sulfurization process were progressed at RTP module, which connected the sulfurization equipment. This unit heats TAA powder at ~130°C and results H₂S gas. Lastly, XPS measurements revealed binding energy shift of $W4f_{5/2, 7/2}$, indicating mostly WO_x converse to WS₂ during the process.

NS-TuP-7 Reversible Electronic Phase Transition in VO₂ Thin Films and Nanostructures, Jun Peng, D. Hensel, Center for Hybrid Nanostructures, Universität Hamburg, Germany; L. Maragno, N. James, Integrated Materials Systems Group, Institute of Advanced Ceramics, Hamburg University of Technology, Germany; C. Heyn, Center for Hybrid Nanostructures, Universität Hamburg, Germany; K. Furlan, Integrated Materials Systems Group, Institute of Advanced Ceramics, Hamburg University of Technology, Germany; R. Blick, R. Zierold, Center for Hybrid Nanostructures, Universität Hamburg, Germany

High-quality vanadium dioxide (VO_2) reveals a phase transition from a dielectric to a metallic state at around 340 K. Despite ongoing discussions regarding the underlying cause of this transition. It has been demonstrated that the complex coupling among lattice, charge, spin, and orbital results in an electronic transition that alters the sample's electrical, thermal, and optical properties at the phase transition. This unique property has led to its utilization exploration in various electronic and optoelectronic applications, including high-temperature thermoelectric materials, thin-film resistors, and optical modulators.

We will present the synthesis and characterization of tailor-made VO2 thin films (2D), nanotubes (1D), and inverse opals (3D) prepared through a combination of thermal atomic layer deposition (ALD, from TDMAV plus water in a custom-built reactor) and subsequent thermal annealing. Temperature-dependent electrical measurements, Raman spectroscopy, and UV-Vis-NIR characterization comprehensively evaluate the electronic phase transition. First, the report will discuss the impact of various parameters during preparation on the thin film quality and the insulator-tometal transition (IMT), including substrates, ALD parameters, and annealing conditions. Afterward, the fabrication of VO2 nanostructured electrical devices will be highlighted, based on the optimized recipe for synthesizing VO₂ thin films with a phase transition temperature of around 335 K, a hysteresis width of approximately 10 K, and a remarkable resistance change of about three orders of magnitude. Specifically, the phase transition in electrically contacted, individual core-shell (Si-VO₂) nanowires is shown. Moreover, we show that the applied voltage can trigger the IMT in such an ALD-based one-dimensional VO₂ device. Finally, we will outline the preparation route for switchable photonic crystals based on inverse VO₂ opals.

Based on the reported results, it can be concluded that ALD of VO₂ holds significant promise for the development of functional, switchable materials in 1D (elongated structures), 2D (thin films), and 3D (inverse opals or bulk-like samples) laying a solid basis for future large-scale applications. Exemplarily, energy-efficient, next-generation nanostructured smart windows are conceivable that can dynamically alter their transmission properties in response to external stimuli, such as temperature, voltage, and current.

NS-TuP-8 Fabrication of 2D-SnS₂ Film Using Atomic Layer Deposition and Hydrogen Sulfide Gas Annealing, *Yeonsik Choi*, S. Song, J. Kim, D. Lee, J. Bae, Y. Lee, H. Jeon, Hanyang University, Korea

2D-tin disulfide (SnS_2) is a material with unique physical, optical, and electrical characteristics that can be used in various optoelectronic devices. In addition, it is possible to fabricate a 2D-SnS₂ material forming a twodimensional layered structure with precise thickness control using atomic layer deposition (ALD). Since 2D-SnS₂ deposited through ALD is based on a low-temperature process, it has the advantage of being applicable not only to silicon substrates but also to flexible substrates such as polyimide (PI) and polyethylene terephthalate (PET). However, since the process is performed at a low temperature, the crystallinity of the material is low and the film quality is relatively poor compared to other processing methods. Therefore, various post-annealing processes that improve the crystallinity of SnS₂ material have been studied, and various aspects such as the crystallinity improvement or the phase transitions were shown depending on the post-annealing atmospheres and temperatures. In this work, we analyzed the crystallinity and grain sizes of 2D-SnS2 deposited by ALD according to the concentration of hydrogen sulfide (H₂S) in a H₂S gas atmosphere at 4.00 and 99.99% and the high crystallinity was shown at $350^\circ C$ with a high concentration of 99.99% H_2S gas. Carrier concentration was also measured and more than 10¹⁸ cm⁻³ was measured at 350°C in the 99.99% concentration of H₂S atmosphere. Comparing to 4.00% concentration of H₂S, pure tetravalent positive states of Sn (Sn⁴⁺) were confirmed through binding energy analysis in the post-annealing in the H₂S atmosphere of 99.99% concentration. In addition, as a result of the postannealing process after deposition on a 4-inch large-area substrate, a high film uniformity and high step coverage (> 98%) on a trench structure wafer were achieved confirming the possibility of future application to the semiconductor industry.

NS-TuP-11 Phase Control of Two-Dimensional Tin Sulfide Compounds Deposited by Atomic Layer Deposition, *Dong Geun Kim*, J. Lee, J. Choi, J. Ahn, Hanyang University, Korea

Two-dimensional (2D) metal chalcogenides have received great attention because of their unique properties, which are different from bulk materials. Among 2D metal chalcogenides, tin sulfide compounds (SnSx) including tin monosulfide (SnS) and tin disulfide (SnS₂) have excellent optoelectronic properties, low melting point, thermal stability, and hydrolytic stability compared to the other representative 2D materials such as MoS₂ and WS₂. Because the electrical property of SnSx is highly dependent on its phase, phase-selective deposition has been required. It has been reported that high-quality SnS_x flakes can be synthesized for chemical vapor deposition (CVD) and sulfurization of metals or metal oxides. However, there is a limitation of applying for next-generation semiconductor devices because of a high process temperature and poor uniformity. Meanwhile, the ALD method based on the self-limiting reaction enables large-area uniformity and conformality over complex-shaped substrates with low growth temperatures. In addition, since the properties of 2D materials are strongly affected by their thickness, the ALD with atomic level thickness control is a suitable deposition technique for 2D materials. In this study, we investigated the phase transition tendency of SnS_x thin films according to deposition temperature and post-annealing atmosphere. SnS₂ phases were dominated at H₂S ambient annealing of relatively low annealing temperature regardless of deposition temperature, according to the increased annealing temperature, the phase transition for SnS₂ to SnS occurred. These phase transitions from SnS₂ to SnS phase were observed at forming ambient gas annealing. In addition, as the annealing pressure decreased regardless of ambient gas, the phase transitions occurred at a lower temperature. Finally, the electrical properties were evaluated by fabricating thin film transistors (TFTs) using optimized conditions of SnS2 and SnS thin films.

NS-TuP-12 Area-Selective Deposition of 2D-MoS₂ using Self-Assembled Monolayer, Jeong-hun Choi, D. Kim, S. Lee, J. Ahn, Hanyang University, Korea

Layered two-dimensional molybdenum sulfide (MoS₂) has attracted great interest for a promising candidate material for opto-electronics and photo sensors applications due to its unique characteristics such as tunable bandgap, high electron mobility and high current on/off ratio. Significant efforts have been placed to apply MoS₂ in industrial fields, leading to significant progress in the deposition method of MoS2. [1],[2] However, patterning technology for MoS₂ remains a challenge. In particular, 2D materials like MoS₂ have extremely thin and weak interlayer bonding due to the absence of dangling bonds, making it difficult to apply traditional topdown patterning approach. Therefore, we demonstrated a new areaselective deposition method for MoS₂ using self-assembled monolayer (SAM). To prevent the degradation of SAM, the deposition of MoS₂ was carried out using a pulsed metal-organic chemical vapor deposition (MOCVD) method, which allowed for the synthesis of high-quality MoS₂ at a low temperature. The growth of MoS₂ was effectively prevented by the SAM patterned using photolithography processes. The selectivity for MoS₂ according to the length of the SAM backbone was investigated using X-ray Fluorescence spectroscopy and Raman measurement. Additionally, the influence of the SAM coating process on the crystallinity and impurity concentration of the MoS₂ film was confirmed using X-ray diffraction and Xray photoelectron spectroscopy. Furthermore, the potential of area-

Tuesday Evening, July 25, 2023

selective deposition of MoS_2 using SAM was demonstrated by fabricating a MoS_2 gas sensor.

Author Index

- A -Ahn, J.: NS-TuP-11, 6; NS-TuP-12, 6 Antonio, E.: NS-TuA-12, 3 Asselberghs, I.: NS-MoA-11, 1 — B — Bae, J.: NS-TuP-8, 6 Bahrami, A.: NS-MoA-13, 1 Benjamin, G.: NS-MoA-11, 1 Bergsman, D.: NS-TuP-1, 5 Bielinski, A.: NS-TuA-14, 3 Blick, R.: NS-TuP-7, 6 Brathwaite, K.: NS-TuA-16, 4 Bright, V.: NS-TuA-12, 3 - C -Cai, R.: NS-TuA-15, 4 Chaney, T.: NS-TuA-12, 3 Chang, C.: NS-TuP-5, 5 Cho, A.: NS-TuP-3, 5 Choe, Y.: NS-TuP-1, 5 Choi, J.: NS-TuP-11, 6; NS-TuP-12, 6 Choi, W.: NS-MoA-16, 2 Choi, Y.: NS-TuP-8, 6 Cott, D.: NS-MoA-11, 1 — D — Dasgupta, N.: NS-TuA-14, 3 David, M.: NS-TuA-13, 3 de Roeck, J.: NS-TuA-13, 3 Delgado, D.: NS-TuA-14, 3 — E — Elam, J.: NS-TuA-11, 3 — F — Faustyn, H.: NS-TuA-14, 3 Fuelling, K.: NS-TuA-14, 3 Furlan, K.: NS-TuP-7, 6 — G — Gayle, A.: NS-TuA-14, 3 George, S.: NS-TuA-12, 3 Greenberg, A.: NS-TuA-12, 3 - H -Hantusch, M.: NS-MoA-13, 1 He, S.: NS-MoA-13, 1 Hensel, D.: NS-TuP-7, 6 Heyn, C.: NS-TuP-7, 6 Hounat, Y.: NS-TuA-13, 3 Hromadko, L.: NS-MoA-14, 1 Hwang, C.: NS-MoA-16, 2

Bold page numbers indicate presenter

— J — James, N.: NS-TuP-7, 6 Jeon, G.: NS-MoA-16, 2 Jeon, H.: NS-TuP-8, 6 Jeon, J.: NS-MoA-16, 2 Jeon, S.: NS-MoA-16, 2 Jhang, Y.: NS-TuP-5, 5 -K -Kanjolia, R.: NS-MoA-15, 1 Kim, D.: NS-TuP-11, 6; NS-TuP-12, 6 Kim, J.: NS-TuP-8, 6 Kim, S.: NS-TuP-3, 5 Kisslinger, K.: NS-TuP-4, 5 Kolíbalová, E.: NS-MoA-14, 1 Krahl, F.: NS-TuP-2, 5 -L-Lee, D.: NS-TuP-8, 6 Lee, J.: NS-TuP-11, 6 Lee, S.: NS-TuP-12, 6 Lee, W.: NS-TuP-4, 5 Lee, Y.: NS-TuP-8, 6 Lehmann, S.: NS-MoA-13, 1; NS-TuP-2, 5 Lin, D.: NS-MoA-11, 1 Lin, Z.: NS-MoA-11, 1 Liu, B.: NS-TuP-5, 5 Luebbert, G.: NS-TuA-16, 4 — M — Macak, J.: NS-MoA-14, 1 Mane, A.: NS-TuA-11, 3 Maragno, L.: NS-TuP-7, 6 McIntee, O.: NS-TuA-12, 3 Mehregan, M.: NS-TuA-16, 4 Miller, M.: NS-MoA-15, 1 Mitzscherling, F.: NS-MoA-13, 1 Morin, P.: NS-MoA-11, 1 -N-Nam, C.: NS-TuP-4, 5 Ngo, T.: NS-MoA-15, 1 Nielsch, K.: NS-MoA-13, 1; NS-TuP-2, 5 Niu, F.: NS-TuA-17, 4 -0-Ong, M.: NS-TuP-1, 5 Ortiz-Ortiz, A.: NS-TuA-14, 3 — P — Park, B.: NS-MoA-16, 2 Pathak, R.: NS-TuA-11, 3

Pavliňák, D.: NS-MoA-14, 1 Peng, J.: NS-TuP-7, 6 Penley, D.: NS-TuA-14, 3 — R — Rennen, R.: NS-MoA-11, 1 Rodriguez Pereira, J.: NS-MoA-14, 1 Rozyyev, V.: NS-TuA-11, 3 Ryu, S.: NS-TuP-3, 5 - s -Santoso, A.: NS-TuA-13, 3 Schram, T.: NS-MoA-11, 1 Segal-peretz, T.: NS-TuA-15, 4 Segal-Peretz, T.: NS-TuA-12, 3 Sergeant, S.: NS-MoA-11, 1 Sherwood, C.: NS-TuA-14, 3 Shevate, R.: NS-TuA-11, 3 Shin, D.: NS-TuP-2, 5 Smets, Q.: NS-MoA-11, 1 Song, S.: NS-TuP-8, 6 Sopha, H.: NS-MoA-14, 1 Stalla, D.: NS-TuA-16, 4 Strzalka, J.: NS-TuA-12, 3 Subramanian, A.: NS-TuP-4, 5 — T — Thalluri, S.: NS-MoA-14, 1 Throm, E.: NS-TuA-16, 4 Tiwale, N.: NS-TuP-4, 5 Toney, M.: NS-TuA-12, 3 - V van Ommen, J.: NS-TuA-13, 3 van Steijn, V.: NS-TuA-13, 3 - W -Wang, J.: NS-TuA-14, 3 Welch, B.: NS-TuA-12, 3 Wijers, N.: NS-TuA-13, 3 Wu, X.: NS-MoA-11, 1 Wyatt, Q.: NS-TuA-16, 4 -Y-Yang, J.: NS-MoA-13, 1; NS-TuP-2, 5 Yoo, C.: NS-MoA-16, 2 Young, M.: NS-TuA-16, 4 -7-Zacatzi, A.: NS-MoA-15, 1 Zazpe, R.: NS-MoA-14, 1 Zheng, R.: NS-TuA-13, 3 Zierold, R.: NS-TuP-7, 6