**Nanostructure Synthesis and Fabrication**  
Room Arteveldeforum & Pedro de Gante - Session NS-Tup

**Nanostructures Synthesis and Fabrication Poster Session**

**NS-Tup-7 In situ Thermal Annealing of ALD Fabricated Pt Nanoparticles and Their Stabilization via Al₂O₃ Overcoating.** E Solano, NCD-SWEET beamline, ALBA Synchrotron Light Source, Spain; J Dendooven, J Feng, Ghent University, Belgium; P Brüner, IONTOF GmbH, Germany; Matthias Minjauw, R Ramachandran, M Van Dalee, K Van De Kerckhove, T Dobbelare, Ghent University, Belgium; A Coati, Synchrotron SOLEIL, France; D Hermida-Merino, ESRF European Synchrotron Radiation Facility, France; C Detavernier, Ghent University, Belgium

The activity and selectivity of supported Pt nanoparticles in heterogeneous catalysis depends on the particle size, shape and coverage [1]. Particle coarsening induced by the often required harsh thermal and chemical conditions during catalytic processes is undesirable since it limits the availability of catalytic sites. Large efforts and resources have been invested to prevent particle coarsening, aiming to prolong the performance and lifetime of nanocatalysts. Among the diverse approaches, particle overcoating via ALD has emerged as a promising method for coarsening prevention while enabling catalyst accessibility through pores and cracks in the overcoat [2]. However, there is a delicate balance in terms of film thickness: a thin overcoat may not be enough for particle coarsening prevention while a thick layer could totally block the surface accessibility for the catalytic application.

Here, we report an in situ synchrotron Grazing Incidence Small Angle X-ray Scattering (GISAXS) study on the thermal stability of supported Pt nanoparticles when overcoated with an Al₂O₃-layer deposited by means of ALD (Figure 1). As model system, we fabricated two distinct sets of samples with equivalent Pt loading but distinct particle coverage [3]. A series of different Al₂O₃ thicknesses were deposited onto the supported nanocatalysts prior to in situ annealing up to 850 °C in an O₂-rich atmosphere. The real-time GISAXS patterns provided a near-continuous feed of information on the evolution of particle size and spacing during the annealing process [4]. In situ data demonstrated that the overcoating methodology requires isolated nanoparticles for an effective stabilization, while stabilization of the closely packed supported nanoparticles turned out to be more challenging, showing the dependence of the minimum overcoat thickness on the initial morphology. Moreover, Pt surface accessibility was evaluated by Low Energy Ion Scattering (LEIS), confirming that the amount of exposed Pt for the coated samples increases during the thermal treatment due to the cracks and pores formation (Figure 2). After a long isothermal annealing, the coated sample with isolated nanoparticles has a larger amount of exposed Pt surface area than an uncoated (and hence coarsened) sample, proving the efficiency of an ALD Al₂O₃ layer as stabilization tool for supported nanocatalysts.


**NS-Tup-11 Surface Functionalization and Atomic Layer Deposition of Metal Oxides on MoS₂ Surfaces, Theodosia Gougousi, J Kropp, C Ataca, University of Maryland, Baltimore County**

Transition metal dichalcogenides (TMD) have been studied for device applications due to their electronic properties and their possession of a bandgap (1.8eV for single layer MoS₂). The fabrication of electronic devices requires deposition of high-quality dielectrics on the hydrophobic TMD surface and remains a challenge.

In this work we will provide examples of TiO₂ and Al₂O₃ Atomic Layer Deposition (ALD) film growth from tetrakis dimethyl amino titanium (TMDAT), trimethyl aluminum (TMA) and H₂O at 100-200°C on exfoliated and Chemical Vapor Deposition (CVD) MoS₂ flakes. We find that the initial surface condition of exfoliated MoS₂ flakes varies greatly due to residual adhesive contamination resulting in large variation in the film morphology. More controlled growth is obtained for CVD MoS₂ flakes but even in this case the surface coverage is thickness dependent for films as thick as 10 nm.

In order to alter the surface energy, seeding with inorganic and organic species have been investigated. Mercaptoethanol (ME) is a small thiol that contains a -SH functional group at one end and -OH termination at the other end. The -SH termination is expected to react with the MoS₂ surface leaving the surface -OH terminated as desired for the onset of the ALD chemistry. Atomic Force Microscopy (AFM) measurements confirm that ME treatment alone does not promote more uniform film growth. Computational studies of the ME-MoS₂ surface interaction using Density Functional Theory (DFT) reveal that ME interacts with the sulfur vacancies almost exclusively. Vacancy density is less than ~5% for as-deposited CVD MoS₂, and depositions result in non-continuous coverage for typical thermal ALD. Argon ion sputtering was used to remove sulfur atoms from the surface and increase the vacancy density up to ~20%. AFM confirms the formation of continuous 6 nm TiO₂ films at 100°C for both ME treated and control samples. DFT calculations reveal that TDMA can interact directly with the vacancy. However, the calculated Density of States for these defect rich surfaces contain gap states that are undesirable for high quality devices. Treatment of defect rich surfaces with ME results in the removal of the gap states which is expected to result in good electrical properties. HAuCl₄ solutions have been also used to seed the surface with -CI terminal species to initiate film growth. XPS data confirm both the presence of Au on the surface and absence of residual chlorine. AFM shows only partial surface coverage for films as thick as 6 nm. A distinct difference in the film morphology is obtained though as a result of this treatment indicating that the film growth mechanism has been altered.

Nanostructure Synthesis and Fabrication
Room Auditorium - Session NS-WeM

2D Nanomaterials by ALD I

Moderators: Tae Joo Park, Hanyang University, Virginia Wheeler, U.S. Naval Research Laboratory

10:45am NS-WeM-1 Exploring ALD 2D Chalcogenides Beyond MoS₂. Miika Mattinen, University of Helsinki, Finland INVITED

Two-dimensional (2D) materials have rapidly emerged as promising materials for a range of applications from microelectronics to sensing as well as energy storage and production. Deposition of 2D materials as high-quality films of controlled thickness on large, temperature-sensitive, and complexly shaped substrates is one of the main challenges hindering industrial applications. ALD can fulfill these requirements, but unlocking the full potential of ALD of 2D materials requires careful examination of precursor chemistry, nucleation, substrates, film characteristics, and device performance.

Much of the ALD research on 2D transition metal dichalcogenides (TMDCs) has focused on semiconducting MoS₂. Lately, some of the efforts have shifted to other 2D semiconductors, which may offer improved charge carrier characteristics (e.g. WS₂ and WSe₂) and lower processing temperatures (SnS₂), for example. So far, little attention has been given to ALD of metallic TMDCs (e.g. NbS₂ and TaS₂), which may be used to complement 2D semiconductors in devices or by themselves in energy storage and production. Furthermore, ALD of selenide and telluride TMDCs is still in its infancy.

Deposition of these “new” 2D chalcogenides by ALD is no simple feat. This talk focuses on finding precursors for a range of 2D chalcogenides from MoS₂ to other semiconductors including HfS₂, SnS₂, WS₂, and ZrS₂ as well as my recent efforts towards metallic sulfide TMDCs. Precursor challenges related to reactivity, thermal stability, and etch reactions will be considered. I will cover some challenges and opportunities related to selenide and telluride TMDCs. I am also going to discuss the stability of the deposited films and deposition of protective layers.

The anisotropic, layered crystal structure of 2D materials affects their growth. The morphology of the grown films in turn affects their properties and performance in applications. The effect of the substrate on morphology, continuity, and crystallinity of 2D films will be discussed through the following three examples. First, the growth of crystalline TMDC films often leads to rough morphology. By depositing amorphous films that are crystallized after deposition, smooth crystalline SnS₂ and WS₂ films can be obtained. Second, deposition of thin and continuous TMDC films is challenging. It will be shown that thinner continuous SnS₂ films can be deposited on sapphire compared to silicon. Third, methods to improve the quality of the films deposited at low temperatures are desired. To this end, I will discuss van der Waals epitaxial growth of 2D chalcogenides.

11:30am NS-WeM-4 Growth of Wafer-Scale Monolayer MoS₂ using Adsorbate-Controlled Atomic Layer Deposition. D Kim, Jae Chan Park, W Kim, Hanyang University, Republic of Korea; J Park, B Shong, Hongik University, Republic of Korea; J Ahn, T Park, Hanyang University, Republic of Korea

Monolayer transition-metal dichalcogenide compounds with two-dimensional (2D) layered structures have attracted considerable attention because of their potential applicability as next-generation active materials for versatile electronic and optoelectronic devices. For industrial application of these materials, a reliable method for well-controlled large-area growth of high-quality material at the wafer scale should be developed. Atomic layer deposition (ALD) of 2D materials has been widely studied for realizing atomically flat monolayer films, but it has limitations regarding the implementation of a perfect monolayer owing to its deposition characteristics.

In this work, we propose a novel chemical route for the deposition of a monolayer MoS₂ film with large-area uniformity at wafer scale using ALD. First, by modulating the precursor injection step, the amount of adsorbed precursor in one cycle can be precisely controlled in a range exceeding the limitation imposed by “typical” ALD reaction. Utilizing such process, we successfully realized (for the first time) a uniform and complete monolayer MoS₂ film using ALD at the wafer scale. The monolayer MoS₂ film exhibited excellent uniformity at the wafer scale, and the luminescence quantum efficiency of a monolayer MoS₂ film deposited via the newly-proposed ALD method was approximately 9 times greater than that of the film formed via conventional ALD. These results indicate that our method can be employed for obtaining complete single layers of various materials or developing high-quality monolayer-scale 2D materials using ALD.
Nanostructure Synthesis and Fabrication
Room Auditorium - Session NS1-WeA

2D Nanomaterials by ALD II

Moderators: Jeffrey W. Elam, Argonne National Laboratory, Hyungjun Kim, Yonsei University, Korea

1:00pm NS1-WeA-1 Atomic Simulation of ALD of 2D Transition-Metal Dichalcogenides, Mahdi Shirazi, E Kessels, A Bol, Eindhoven University of Technology, Netherlands

Extensive research has been done during the last decade to unravel the remarkable electronic properties [1], [2] of two dimensional transition-metal dichalcogenides (2D-TMDs) in the monolayer regime. In spite of their astounding electrical properties, these material systems are not ready yet for replacing Si based materials for future nanometer-sized electronic devices. One key challenge is the integration of these materials in bottom-up processes at low temperature (usually < 500 °C) into the semiconductor manufacturing flow. Horizontal growth at wafer scale with a large grain size (typically 1x1 μm) is required for nano-electronic devices [3]. The cyclic process of atomic layer deposition (ALD) [4] with tight control over the chemical reactions shows promise as such a bottom-up process. The chemical reactions of ALD are self-limiting and are designed to proceed only at the surface. In this contribution, we have employed density functional theory (DFT) to provide fundamental insight into the reaction mechanisms of the MoS₂ growth. We have studied the deposition of MoS₂ that is initiated by the exposure of metal precursor Mo(NMe₆)₂(NbBu₅)₂ (C₁₈H₂₅N₅Mo₂) to the SiO₂ surface and then followed by exposure of H₂S/H₂ as co-reagent in the second pulse [5]. In this so-called hetero-deposition, the involved chemical reactions during ALD lead to the formation of a buffer layer at the surface of SiO₂ [6]. After formation of this buffer layer, ALD enters into the steady-growth regime (also called homo-deposition). In the steady growth regime, vertically or horizontally aligned MoS₂ structures grow in a layer-by-layer fashion. The calculated reaction energies and activation energies indicate that the reaction kinetics in the hetero-deposition are slower than reaction kinetics in homo-deposition. Artificial intelligence is used to generate an efficient interatomic potential using the calculated energies and forces of configurations obtained by DFT. The generated interatomic potential will be used for larger scale simulations to provide further fundamental insight into the deposition of MoS₂ by ALD.


1:45pm NS1-WeA-4 ALD of MoSe₂ using New Precursors, Raul Zazpe, University of Pardubice, Czech Republic; R Krumpolec, Brno University of Technology, Czech Republic; J Charvat, L Hromadka, H Shopa, M Motola, M Krabal, F Bures, J Macak, University of Pardubice, Czech Republic

2D semiconductor transition metal dichalcogenides (TMDs) have attracted considerable attention due to their layered structure, suitable band gap for visible light absorption, high carrier mobility, electrochemically active unsaturated edges and relatively good stability against photocorrosion [7]. Recently, 2D MoSe₂ has been gaining considerable interest due to its higher electrical conductivity as compared to MoS₂, its wider inter-layer distance (~0.66 nm), narrow bandgap (1.33–1.72 eV), high resistance to photocorrosion, high surface area layer, electrochemically active unsaturated Se-edges and close to zero Gibbs free energy edges for hydrogen adsorption. These properties are promising for different applications of MoSe₂, including hydrogen evolution [2], photocatalysis [3] and Li-ion batteries [4]. However, their low light absorption efficiency, recombination issues of the photogenerated electron–hole pairs and slow charge transfer of the intrinsic semiconducting 2H-phase are a handicap. An efficient strategy to surmount these intrinsic limitations are hybrid nanostructures using conducting supporting materials. In this regard, anodic TiO₂ nanotubes (TNTs) are excellent photoactive supporting material providing a high surface area, unique directionality for the charge separation, and highly effective charge collection. Accordingly, we present anodic TiO₂ nanotubes homogenously decorated with MoSe₂ nanosheets by atomic layer deposition (ALD). In parallel, we address the current scarcity of convenient ALD Se precursors by the synthesis a set of new selenium precursors - alkyliyli (R₆Si):Se and alkylin (R₆Sn):Se, and cyclic silylenes compounds. Those Se precursors were extensively characterized and their reliability as ALD Se precursors was confirmed by ALD. Several uniform nanosheets with exposed promising results to be convenient ALD Se precursor as will be presented in the synthesis. The presentation of the MoSe₂ nanosheets and their composites with TiO₂ NTs, their physical and electrochemical characterization, and encouraging results in electrochemical characterization, hydrogen evolution reaction (HER) and photocatalysis will be presented and discussed.

2:00pm NS1-WeA-5 Low Temperature Creation of Layered-MoS₂ Thin Films on Large Area High Aspect Ratio Substrates, Anil Mane, D Choudhury, S Letourneau, J Elam, Argonne National Laboratory

Thin layers of two dimensional (2D) materials mainly transition metal dichalcogenides (TMDs) and more specifically ultra-thin-layered-MoS₂ semiconductor possess exceptional properties such as electrical, optical, magnetic, mechanical and chemical properties. This allows the exploration of internal quantum degrees of freedom of electrons and their potential for use in semiconductor microelectronics, optoelectronic, energy, and sensor and detector applications. These exciting results are being achieved mostly by using exfoliation of flakes from bulk MoS₂ crystal. However, the biggest challenge in realizing TMDs full potential has been the lack of scalable material synthesis methods. ALD thin films with high uniformity, conformality and interfacing with other materials such as oxides, Si, and its process compatibility.

Among the various thin film deposition methods, atomic layer deposition (ALD) offers the best combination of precisely controlled layer-by-layer thin film growth at low temperature with very high conformality on complex substrates. Here we will present the growth of layered-MoS₂ thin films. To grow high quality layered-MoS₂ thin films, we have developed an ALD-based two step processing approach [1]: firstly the growth of well controlled ultra-thin layer of Mo metal using ALD followed by the sulfurization of the ALD Mo layer at various temperatures. This two-steps processing results in high quality layered-MoS₂ thin films on large substrates. For the Mo ALD process we used molybdenum hexafluoride (MoF₆) and SiH₄ precursors. We used in-situ QCM measurements to study interfacial and nucleation effects in the formation of continuous ultra-thin metal layer of Mo. The composition of both the ALD Mo and the layered MoS₂ layers was determined by X-ray photoelectron spectroscopy (XPS). Further, cross-sectional transmission electron microscopy (TEM) was performed to confirm the formation of layered MoS₂ on high aspect ratio trenches and Raman analysis to verify the signature of E₂g blue shift and A₁g red shift in the MoS₂ structure. In this presentation we will discuss the details of the two-step thin film growth process for creating layered MoS₂ layers via ALD Mo and subsequent sulfurization as well as the properties of the MoS₂ films.


2:15pm NS1-WeA-6 Gas Sensing Characteristics of Mo₆W₁₋₅S₆ Synthesized by Atomic Layer Deposition, Inkyu Sohn, Y Kim, M Lee, J Park, H Kim, Yonsei University, Republic of Korea

Two-dimensional Transition metal dichalcogenide (2D TMDC) has been attracting great attention as gas sensing materials with high sensitivity in room temperature. [1] Because of this characteristic, 2D TMDC gas sensor could overcome the oxide-based semiconductor which need heating for gas sensing. Therefore, various 2D TMDC gas sensor studies have been ongoing. Recently, it has been shown that the gas sensor property could be improved through the changing the composition in WS₂:Seₓ → alloy. [2]

Here we report a synthesis method of Mo₆W₁₋₅S₆ alloys for gas sensor by atomic layer deposition. Layer controlled 2D MoS₂ and WS₂ were synthesized with Mo(CO)₆, W(CO)₆ and H₂S as precursors and reactant. For the first time, we systematically modulate the composition of Mo₆W₁₋₅S₆ alloys by changing the configuration of low-temperature ALD super-cycles. AFM and Raman spectroscopy results of Mo₆W₁₋₅S₆ demonstrate that the thickness of alloy is accurately controlled by ALD. Also XPS results confirmed that composition of alloy is precisely controlled by ALD supercycles. Furthermore, gas sensors fabricated by Mo₆W₁₋₅S₆ alloys were evaluated for NO₃ gas. It showed response time and recovery characteristics of Mo₆W₁₋₅S₆ alloy gas sensor is dramatically enhanced.
Nanostructure Synthesis and Fabrication
Room Auditorium - Session NS2-WeA

Nanomaterials by ALD - Nanoparticles and ALD on Polymers
Moderators: Jeffrey W. Elam, Argonne National Laboratory, Hyungjun Kim, Yonsei University, Korea

3:00pm NS2-WeA-9 Visualizing the Nucleation of ALD on Polymers, Laura Asteroe, Ghent University - IMEC, Belgium; P Esbah Tabaei, Ghent University, Belgium; D Schaubroeck, M Oop de Beeck, Ghent University - IMEC, Belgium; R Morent, Ghent University, Belgium; H De Smet, Ghent University - IMEC, Belgium; N De Geyter, Ghent University, Belgium

Polyimides are vastly used in the microelectronics industry as they can withstand the conditions of many processes due to their mechanical robustness, and thermal and chemical stability. A specific polyimide (PI) (2611) has been used as packaging material for flexible implantable medical devices and electrodes due to its relatively good water barrier properties and biocompatibility. Yet, it cannot provide the necessary hermeticity for long term implantation. Therefore, this PI can be combined with metal oxide ALD layers (SiOx, AlOx, TiOx or HfOx) to develop ultra-hermetic barriers (WVTR = 10^-8 g/cm²day). To characterize the interaction between the PI and the ALD layers it is fundamental to understand the nucleation of the ALD layer on the polymer. These are commonly studied by quartz microbalance, ellipsometry, FTIR, or XPS, all of which provide valuable information but do not allow visualizing the nucleation behavior during the initial stages of ALD growth until full closure of a layer on polymers. In this contribution, a new qualitative method to study this is proposed and applied to the Al₂O₃ and HfO₂ ALD deposition on PI. The deposited metal oxide acts as a hard mask during PI reactive ion etching (RIE). This results in local features of the deposited ALD being visible in FEG SEM images. The samples were also analyzed by XPS to verify the Al or Hf content present on the surface. The proposed method produces repeatable results, and by increasing the exposure time of the RIE it was confirmed that the deposited Al₂O₃ or HfO₂ was not damaged by the etching step. As expected, the images taken and the XPS results showed a faster nucleation and growth of a fully closed layer of the AlOx compared to HfOx. In both cases an island-coalescence type of nucleation and growth was observed. First, islands were formed on the surface of PI, growing into filamentous structures that progressively coalesced into meshes, which became denser until eventually a closed layer was formed. The Al or Hf content measured by XPS presented an equivalent increase behavior. The content of Al or Hf was low and constant at the beginning, corresponding to the island stage. It progressively increased as the mesh structures were formed and became denser until it stabilized at a constant value. Herein, a qualitative method to visualize the nucleation and growth of a full ALD layer on polymers was introduced. It is not presented as an alternative to the available techniques but rather as a complementary tool.

3:30pm NS2-WeA-11 Pt-Doped InO₃ Thin Films: Control of the Chemical State and Structure via ALD, Ranjith K. Ramachandran, M Filez, Ghent University, Belgium; E Solano, NCD-SWEET beamline, ALBA Synchrotron Light Source, Spain; R Ramachandran, M Miniaou, M Van Daele, Ghent University, Belgium; D Hermida-Merino, ESRF European Synchrotron Radiation Facility, France; A Coati, Synchrotron SOLEIL, France; C Detavernier, J Dendooven, Ghent University, Belgium

It is well known that noble metal addition to metal oxides improves the optical and electronic properties of the oxides. Depending on the targeted application of the resulting material, another mode of addition is demanded. While noble-metal doped semiconductor metal oxides show improved catalytic and gas sensing properties, metallic nanoparticles (NPs) embedded in metal oxides are used as charge storage materials in single-electron memory devices. In any case, accurate tuning of the dopant level/NP’s size is crucial to achieve an optimal performance. Here, a novel atomic layer deposition (ALD)-based method for doping Pt into InO₃ is demonstrated. We combine alternating exposures of Pt and InO₃ ALD processes in a single “supercycle” followed by supercycle repetition leading to multilayered nanocomposites (Figure 1). The resulting samples of Pt embedded in an InO₃ matrix are characterized chemically and structurally using a variety of X-ray-based techniques, such as X-ray fluorescence (XRF), X-ray diffraction (XRD), X-ray absorption spectroscopy (XAS), grazing-incidence small-angle X-ray scattering (GISAXS), and high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM), combined with energy dispersive X-ray spectroscopy (EDS).

The proposed method enables controlled doping of Pt/PtO₃ species inside the InO₃ matrix with the ability to fine-tune their chemical and structural properties. For example Figure 2 shows the results from a series of samples (samples B1 – B6) where the number of Pt ALD cycles was varied from 1 to 30 while keeping the number of InO₃ cycles at 30. XAS and XRD shows that a few Pt ALD cycles lead to the formation of oxidized Pt species due to their highly dispersed nature. GISAXS and HAADF-STEM, combined with EDXS, show that Pt is evenly distributed in the InO₃ matrix without the formation of clusters. For a larger number of Pt ALD cycles, the oxidation state gradually evolves toward fully metallic, and metallic Pt clusters are obtained within the InO₃ matrix.

In addition to giving valuable information on the dopant distribution and its chemical state, which provides strategies for fine-tuning the properties of doped metal oxides, this approach allows characterization of the nucleation stages of metal ALD processes by freezing those states through embedding and stacking them multiple times. This allows the use of XAS and other techniques to study even a single-metal ALD cycle, without the need for the use of high-surface-area supports.

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