On Demand

Nanostructure Synthesis and Fabrication
Room On Demand - Session NS2
Nanotubes, Nanowires, Nanopores

NS2-1 Atomic Layer Deposition for Modification of Various 1D Nanomaterials*, R. Zazpe, H. Sopha, M. Motola,* Uni Pardubice, Czechia*; M. Rihova,* Brno University of Technology, Czechia*; **Jan Macak**,* Uni Pardubice, Czechia

One-dimensional nanomaterials – materials with one dimension outside the nanoscale, further noted as 1D NMs – represent a class of very important nanomaterials with continuously increasing importance. Due to their intrinsic features, unique properties and diversity of functionalities, they count among the most widely studied materials nowadays. While considerable research efforts have been spent to synthesize various 1D NMs (e.g. nanopores, nanotubes or nanofibers), limited efforts have been devoted to surface modification and property tailoring of these materials.

However, it is their surface that comes into direct contact with various media (air, gases, liquids, solids) and influences the reactivity, stability and biocompatibility of these materials. The surface and aspect ratio (defined as their diameter to length ratio) influence the performance of these materials in various applications. Considering these facts, it is more relevant to tailor the surface of these materials and to be able to influence their properties and reactivity at the nanoscale, rather than to deal with tailoring their own bulk material composition.

The focus of this presentation is on the modification of two types of 1D nanomaterials – nanotubes and nanofibers. Numerous techniques can be utilized for this purpose, such as for example wet chemical or physical deposition techniques. However, it is only the Atomic Layer Deposition (ALD) that is capable of really uniform and homogenous coating of these 1D nanomaterials, in particular those of very high-aspect ratio.

The presentation will be mainly focused on modification of TiO_2 nanotube layers and various nanofibers of different aspect ratios via ALD.

Experimental details and some very recent application examples [1-9] and structural characterizations of these modified materials will be discussed.

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Nanostructure Synthesis and Fabrication
Room On Demand - Session NS3
2D Nanomaterials by ALD (including Transition Metal Dichalcogenides)

NS3-1 2D Core-Shell Quantum Dots Grown by ALD*, **Jeff Schulpen**, M. Verheijen, E. Kessels, V. Vandalon, A. Bol,* Eindhoven University of Technology, Netherlands

Two-dimensional transition metal dichalcogenides (e.g. MoS_2, WS_2) have fascinating optical and electronic properties that make them promising materials for use in next-generation devices. In particular, 2D quantum dots (i.e. nanoparticles of 2D materials) have received great interest in recent years for their effectiveness in catalysis as well as opto-electronic and energy-storage applications [1]. Furthermore, the versatility of 2D materials is greatly enhanced by the fact that they can be assembled with atomically sharp interfaces, resulting in heterostructures that benefit from the combined functionality of multiple 2D materials as well as completely new material properties [2]. Applying heterostructure methods to 2D quantum dots could result in core-shell nanoparticles which combine the useful properties of 2D quantum dots with the versatility of heterostructures. However, little is known about such structures, as most of the literature is aimed at single-material quantum dots.

In this work, we show that 2D monolayer core-shell quantum dots can be grown by atomic layer deposition. Using a supercycle approach, we grow crystalline nano-scale monolayer flakes of MoS2 which are subsequently epitaxially bordered by WS_2. This is achieved by exploiting the reactive edge sites of these materials, by switching from MoS_2 to WS_2 deposition before a closed monolayer is formed. Notably, this method can be extended to the growth of more elaborate structures (e.g. core-shell-shell), whereby the diameter of the core and shell(s) can be straightforwardly and accurately controlled through the number of ALD cycles. In practice, the size control of the grown core-shell structures is limited by the nucleation density of the ALD process, which is linked to the density of reactive sites on the substrate [3,4]. In order to enable the growth of larger and more elaborate structures, we employ strategies to reduce the nucleation density. The growth is investigated in-situ using spectroscopic ellipsometry, and the grown structures are characterized by AFM and TEM, as well as Raman and PL spectroscopy. We expect our technique of growing 2D core-shell quantum dots to extend to other 2D material systems, thereby establishing an important new method of tailoring the properties of 2D quantum dots for applications in catalysis, opto-electronics and energy-storage.

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NS3-2 2D Molybdenum Dichalcogenides Family by Atomic Layer Deposition*, **Raul Zazpe**,* University of Pardubice, Czech Republic*; R. Krumpolec,* Masaryk University, Czech Republic*; J. Charvot, L. Hromadko,* University of Pardubice, Czech Republic*; H. Sopha,* University of Pardubice, Czech Republic , Czechia*; M. Motola, F. Bures, J. Macak,* University of Pardubice, Czech Republic

2D semiconductor transition metal dichalcogenides have attracted considerable attention due to their layered structure, suitable band gap, electrochemically active unsaturated edges and relatively good stability against photocorrosion. These properties result promising for different applications including, Li-ion batteries, photocatalysis and hydrogen evolution reaction (HER). Apart from the widely studied 2D MoS2, 2D selenide and telluride equivalents, MoSe2 and MoTe2, have recently gained considerable interest due to their higher electrical conductivity, wider inter-layer distance and narrower bandgap as compared to MoS2, high surface area and close to zero Gibbs free energy edges for hydrogen adsorption. Unlike sulfide dichalcogenides, the lack of Se and Te precursors have prevented the synthesis of selenide and telluride dichalcogenides by ALD. In order to surpass such impediment, we present a set of novel in-house synthesized Se and Te compounds, which were successfully combined with commercial Mo precursor to synthesize MoSe2 and MoTe2 by ALD [1-5]. The as-deposited ALD MoSe2 and MoTe2 on substrates of different nature were extensively characterized by different techniques, which confirmed the chemical composition and revealed the growth of 2D flaky nano-crystalline MoSe2 and MoTe2. In parallel, MoSe2 and MoTe2@TiO2 nanotube layers (TNTs) heterostructures were fabricated in a simple and fast fashion to explore and exploit the MoSe2 and MoTe2 photo- and electrocatalytic properties. TNTs act as excellent photoactive supporting material providing a high surface area, unique directionality for charge separation, and highly effective charge collection.

The presentation will introduce and describe the synthesis of the 2D Mo dichalcogenide family, the corresponding physical and electrochemical characterization and encouraging results obtained in HER [4,5], photocatalysis [4-6] and Li-ion batteries [7].

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NS3-3 Atomic Layer Deposition of Ultrathin Tungsten Oxide Films for 2D WS_2 Synthesis*, **Maxim Kozodaev**, R. Romanov, A. Markeev,* Moscow Institute of Physics and Technology, Russian Federation

Tungsten oxide attracts great attention as a functional layer of electrochromic [1] or resistive memory devices [2], but also as a source for the synthesis of two-dimensional ultrathin WS_2 nanosheets, which can serve as a channel material in the post-silicon FET technology [3]. In this regard, ALD utilization for the tungsten oxide growth is highly preferable since it allows the best thickness control and reproducibility over other deposition techniques, which is crucial for mass-production. Previously it has been reported that radical-enhanced ALD also allows precise oxygen deficiency control in WO_x films [4].

In this work, we modify the previously reported $WH_2(Cp)_2$-based ALD process by the oxygen source exchange to ozone. This process showed a clear saturation behavior on both reactants, good deposition uniformity, and higher oxygen deficiency level, which was monitored by in-situ X-Ray photoelectron spectroscopy to exclude film degradation under ambient conditions. The V_o presence was concluded from the visible electronic states formation in the bandgap, an increase in the concentrations of tungsten in the W5+ state and oxygen in the non-lattice state. An increase in the V_o concentration was accompanied by a significant film resistivity decrease and an abrupt change in the crystalline structure, which was revealed by XRD analysis. The early stages of the suggested WO_x ALD process were studied by angle-resolved XPS and the moment of film continuity was determined. The subsequent sulfurization allowed its' effective conversion to WS_2 nanosheets with the terrace-terminated structure. Recently it was shown that preliminary seed WO_x film hydrogen treatment allowed to significantly change WS_2 morphology and improve its' electrical quality [5]. Noteworthy, exactly ultrathin WS_2 layers are of particular interest, therefore, the seed WO_x thickness influence on the resulting WS_2 morphology was also investigated. In particular, such a technique allowed us to obtain the continuous WS_2 nanosheets with a thickness down to 3-4 monolayers. The obtained results expand the opportunities horizon of the 2D materials by the possibility of a fab-oriented production technique.

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NS3-6 Wafer-Scale Synthesis of Transition Metal Dichalcogenide Thin Films by ALD-Based Technique Towards Nanoelectronics and Optoelectronics Applications*, **Hao Zhu**, Z. Gu, T. Zhang, H. Liu, L. Chen, L. Ji, Q. Sun,* Fudan University, China

Two-dimensional (2D) transition metal dichalcogenides (TMDs) are attracting growing interests in recent years due to their unique electronic properties even with thickness at atomic scale. Though tremendous progress has been made in the understanding and implementation of the physical properties of TMDs in advanced micro-/nanoelectronic devices through conventional fabrication methods like mechanical exfoliation or chemical vapor deposition (CVD), the large-scale device integration for system-level applications have been severely bottlenecked by the lack of effective synthesis approaches to achieve wafer-scale, uniform, crystalline and stoichiometric TMD films.

Atomic layer deposition (ALD) is a surface-controlled film fabrication and can provide a promising route towards the synthesis of high-quality TMD thin films since the ALD approach follows the layer-by-layer deposition mechanism. As compared to other synthetic methods like CVD, ALD can enable precise thickness control on atomic scale and excellent film uniformity as well as good stoichiometry and crystallinity with proper annealing steps. Here, we have developed ALD-based synthesis approaches to prepare wafer-scale MoS_2 and WS_2 TMD thin films. Non-toxic $MoCl_5$, WCl_5 and hexamethyldisilathiane (HMDST) are used as precursors. Film characterizations have confirmed the wafer-level uniformity and good crystallinity by annealing in sulfur atmosphere. The field-effect transistor (FET) device arrays fabricated on the wafer-scale TMD film have shown excellent homogeneity in electrical performance, which provides good platform for further integration. In our work, we have fabricated optoelectronic device arrays as well as inverter, NAND, NOR, AND, and OR logic gates which have shown robust and repeatable logic functions. This has paves solid basis for the system and circuit-level applications.

It should also be noted that we have been designing and optimizing a high-temperature ALD process to grow crystalline TMD thin films eliminating the high-temperature annealing step in the previous flow. Preliminary experimental results have been achieved demonstrating satisfactory film quality, and the FET devices fabricated on it can exhibit repeatable switching performance with on/off ratio over 10^3. We believe that this can be a more encouraging synthetic method to prepare wafer-scale TMD thin films for practical electronic applications.

Nanostructure Synthesis and Fabrication
Room On Demand - Session NS4
ALD on 2D Related Materials and Devices

NS4-1 Recovery Enhancement of Al_2O_3 Functionalized MoS_2 Gas sensor by Atomic Layer Deposition*, **Inkyu Sohn**, S. Wi, Y. Kim, M. Kim, H. Yoon, S. Jung, H. Kim,* Yonsei University, Korea

Two-dimensional (2D) transition metal dichalcogenides (TMDs) which possess large surface-to-volume ratio have been widely used for room temperature gas sensing applications. [1] However, due to its intrinsic defect or vacancies on TMD surface, incomplete recovery of TMD gas sensors hinder the realization of reliable and repeatable use of 2D TMD gas sensors. [2] Here, we demonstrate improvement of recovery rate of TMD gas sensors by covering TMD surfaces' defect or vacancies with Al_2O_3 using atomic layer deposition. Raman, AFM, XPS data have been showed. Especially, SEM data have showed that Al_2O_3 is partially covered on grain boundaries or defects of MoS_2 which is favorable for enhancing recovery performance of TMD gas sensors. The pristine MoS_2 gas sensors show 74 % of recovery rate in the case of NO_2 sensing. However, the recovery rate of the Al_2O_3 functionalized MoS_2 gas sensors improved up to 96 %. Our proposed method shows promising strategy for improving recovery rate of 2D TMD gas sensors.

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Nanostructure Synthesis and Fabrication
Room On Demand - Session NS5
ALD on Polymer Materials

NS5-1 ALD of In_2O_3 in PMMA: Resolving the Atomic Structure of Sequential Infiltration Synthesized Clusters*, X. He, R. Waldman, S. Darling, D. Tiede, **Alex Martinson**,* Argonne National Laboratory

Sequential infiltration synthesis (SIS) is a route to the precision deposition of inorganic solids in analogy to ALD but occurs within a soft material template. SIS has enabled exquisite nanoscale morphological complexity in several oxides through selective nucleation in block copolymers templates. However, the earliest stages of SIS growth remain unresolved, including the atomic structure of nuclei and the evolution of local coordination environments, before and after polymer template removal. We employed In K-edge extended X-ray absorption fine structure and atomic pair distribution function analysis of high-energy X-ray scattering to unravel the structural evolution of InO_xH_y clusters inside a poly(methyl methacrylate) (PMMA) host matrix. Early SIS cycles result in InO_xH_y cluster growth with high aspect ratio, followed by the formation of a three-dimensional network with additional SIS cycles. That the atomic structures of the InO_xH_y clusters can be modeled as multinuclear clusters with bonding patterns related to those in In_2O_3 and $In(OH)_3$ crystal structures suggests that SIS may be an efficient route to 3D arrays of discrete-atom-number clusters. Annealing the mixed inorganic/polymer films in air removes the PMMA template and consolidates the as-grown clusters into cubic In_2O_3 nanocrystals that form porous In_2O_3 solids, the morphology of which also depends on cycle number.

NS5-2 Understanding and Controlling Polymer-Organometallic Precursor Interactions in Sequential Infiltration Synthesis*, Inbal Weisbord*, *N. Shomrat, R. Azoulay, A. Kaushansky, T. Segal-Peretz,* Technion - Israel Institute of Technology, Israel

With the emergence of sequential infiltration synthesis (SIS) as a prominent method for growth of inorganic materials inside polymeric structures, it becomes essential to understand the mechanism of precursor diffusion and reaction inside polymers, in order to expand the scope of SIS, and allow growth in thicker specimen, under more economic conditions.

This study uses a combination of density functional theory calculations and *in-situ* microgravimetric measurements to shed light on the equilibrium interaction between the polymers polymethyl methacrylate (PMMA) and poly-2-vinyl pyridine (P2VP) and the organometallic precursor trimethyl aluminum (TMA). The relationship between temperature, diffusion and reaction is probed, to reveal that while diffusion increases with temperature, the strength of reaction decreases. In low temperatures the strong reaction forms a low diffusivity region at the top of the film and inhibits further growth. At high temperatures, diffusion is high, but poor reaction strength prevents the precursor from binding in the film. The study finds the balance point, which is the optimum point that maximizes both factors, for each polymer. SIS dynamics, prior to reaching equilibrium, are probed using *in-situ* microgravimetric measurement, as well, enabling precise control over AlO_x growth in PMMA and P2VP, as corroborated by electron microscopy. The knowledge gained from this thorough analysis is then used to design a process of TMA growth inside self-assembled polystyrene-*block*-P2VP in a manner that both preserves the self-assembly and allows for significant AlO_{x-} growth.

NS5-3 Atomic Layer Deposition of Al_2O_3 and TiO_2 on Polydimethylsiloxane*, Albert Santoso*, *B. van den Berg, V. van Steijn, J. van Ommen,* TU Delft, Netherlands

Atomic layer deposition (ALD) on polymeric substrates is attracting increasing attention due to potential applications in OLEDs, food packing, and biomaterials. By depositing a very thin film of metal oxide, the surface properties, such as wettability, and overall performance of the material can be adjusted without losing the unique bulk properties. However, due to the inherent porous structure and competing precursor reaction on the polymeric branch, the deposition of metal oxide on polymers does not follow the ideal ALD mechanism. The subsurface growth and minimal surface diffusion lead to a non-conformal layer and subsequently different final properties. In this study, we focus on the surface modification of polydimethylsiloxane (PDMS) which is widely used material to fabricate microreactor and lab-on-a-chip devices. We have investigated the use of ALD of alumina and titania on PDMS using two different ALD reactors (vacuum and atmospheric) at various operating conditions (temperature, substrate pretreatment). X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM) and scanning electron microscopy (SEM) have been used for material characterization, while contact angle hysteresis measurements have been used for approximating the surface energy stability over time. Results indicate that at high temperature (120°C), micro-sized cracks and buckles are formed due to a difference in thermal expansion coefficient of metal oxide and PDMS, which density are reduced as the temperature goes down. When comparing the effect of oxidizing precursor, thermal ALD of metal oxide on PDMS leads to severe infiltration of precursors, and subsequently subsurface growth up to micron range, which have little influence on the surface wetting property. On the other hand, low temperature plasma-assisted vacuum ALD results into a surface growth, suggesting the growth kinetic is more dominant than the precursor diffusion, although nano-sized cracks and non-conformity were also still observed, which end up with certain hydrophobic recovery. The recovery rate is reduced by using atmospheric ALD. Further XPS analysis shows that there are some elements of ligand components from the precursor that may remain trapped due to incomplete purging, which might act as a pinhole-filler in subsurface infiltration and surface layer growth, leading to higher stability in surface energy. This study gives insight in utilizing thin films grown by non-ideal ALD as buffer layers for robust modification of polymeric substrates, which could be applied in not only controlling fluid behavior but also obtaining the desired surface property.

NS5-6 Modified 3D Printed Architectures: Effects of Coating and Infiltration by Alumina on ABS*, Atilla Varga*, *B. Nwokolo, P. Gordon, S. Barry,* Carleton University, Canada

In recent years 3D printing has gained enormous popularity thanks to its affordability, accessibility, ease of use, and the ability to easily employ a variety of polymer materials. These potential polymer substrates are highly tunable in flexibility and strength which make them the ideal for printing filaments, they can incorporate metal powders, wood fibers, carbon fibers, etc.[1] The integration of 3D printing and industrial nanoscale processes such as ALD will have a significant impact in the development of advanced 3D printed architectures leading to a wide array of applications.

ALD modification of 3D printed structure can be applied in nanoparticle growth, which has applications in low-cost high surface area catalysts. Both thin film coatings as well as precursor infiltration have already been studied in chemical resistant polymer coatings, conductive polymer networks, and various other applications of interest in electronics, sustainable materials and manufactured products.[2]

Previously ALD has been used to deposit alumina thin films on various types of polymers.[3] This work will expand the deposition of alumina onto 3D printed, of one of the most commonly available 3D printed substrates: acetonitrile butadiene styrene (ABS). The observed effects of either coating or infiltration of alumina on the polymers' physical properties such as melting point, glass transition temperature, thermal decomposition, and conductivity will be studied. The effects of coating versus infiltration will be contrasted to demonstrate the extent to which these characteristics can be tuned.

Scanning electron microscopy (SEM) and energy dispersive X-Ray spectroscopy (EDX) will be used to examine the modified polymer structures (Figure 1). Differential scanning calorimetry (DSC), thermogravimetric analysis (TGA) and conductivity probe measurements will be used to observe the physical property changes of the treated polymer compared to the untreated.

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Nanostructure Synthesis and Fabrication
Room On Demand - Session NS6

Nanostructures Synthesis and Fabrication Poster Session

NS6-1 Synthesis of Silicon Carbide Thin Films by Post-Processing of Molecular Layer Deposition (Mld) Polyamide Films on Silicon*, Rustam Amashaev*, Dagestan State University, Russian Federation*; I. Abdulagatov,* Dagestan State University , Russian Federation

Silicon carbide (SiC) is a technologically important material that has many industrial applications. SiC films on Si substrates have been used to fabricate transistors, optical waveguides and components for quantum computers. In addition, SiC films have been used to obtain another technologically significant material – graphene. There are several primary ways to deposit SiC films: physical and chemical vapor deposition (PVD and CVD). Although these methods can deposit good quality films, they cannot fulfill all the stringent requirements, in terms of film conformality, thickness control, and cost effectiveness, needed to manufacture modern microelectronics devises.

In this work, we studied alternative approach for controlled synthesis of epitaxial quality SiC thin film. This method based on pyrolysis of MLD polyamide film deposited on single crystal Si substrate (see Fig.) [1]. MLD allows to deposit highly conformal organic thin film with precise thickness control. Consequently, it might be possible by controlling the parameters of the initial MLD film accurately control the thickness and uniformity of the SiC film.

MLD polyamide films were deposited at 120 ^{o}C by thermally stimulated surface reactions between trimesoyl chloride (TMC) and 1,2-ethylenediamine (EDA). *In situ* QCM monitoring of the MLD process showed linear increase in mass with the number of cycles. The polyamide film growth rate on Si(111) was 18.5 Å/cycle. After deposition, pyrolysis was conducted at temperatures between 1000 and 1300 ^{o}C and pressure of ~10^{-7} Torr. Number of *ex situ* characterization methods, such as, scanning electron microscopy (SEM), x-ray diffraction (XRD), Raman, energy-dispersive x-ray spectroscopy (EDX), and reflection high-energy electron diffraction (RHEED) were employed to analyze samples after pyrolysis. These techniques confirmed that after heat treatment single crystal b-SiC (3C-SiC) thin films have been synthesized.

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NS6-2 Fabrication of Free-standing Three-dimensional Structures by Spatial Atomic Layer Printing*, **Philipp Wiesner,** I. Kundrata,* ATLANT, Germany*; S. Tymek, M. Barr,* Friedrich-Alexander-University Erlangen-Nürnberg (FAU), Germany*; M. Plakhotnyuk,* ATLANT, Denmark*; J. Bachmann,* Friedrich-Alexander-University Erlangen-Nürnberg (FAU), Germany

One of the most common processes to produce microelectronics is optical lithography. It combines deposition by different thin film techniques including CVD or ALD with etching of defined structures using masks.This multistep procedure is time-consuming and wasteful of material. Spatial Atomic Layer Printing offers an alternative for the direct production of defined structures and therefore for components in microelectronics.

In this study, free-standing three-dimensional bridges are produced using the atomic layer 3D printer, developed by ATLANT 3D Nanosystems, that provides direct atomic layer writing process. For this purpose, two crossing lines are deposited from a support and a print material, subsequently the support material is removed via wet chemistry etching processes. To ensure an intact bridge, different material combinations of platinum, titanium dioxide, silicon dioxide and zinc oxide are used and post-processed by different etching methods. In particular, the materials used and the type of etching play an important role in producing freely standing structures. Important aspects in the preparation of the component are the influence of the etchant on the materials, the temperature used, the surface tensions of the etchant and the cleaning agent as well as the mechanical stress during the process.

The most promising material combination during the test series is a printing material of titanium dioxide deposited with TTIP and H2O and a support material of zinc oxide deposited with diethylzinc and H2O. To ensure the gentlest possible etching process, the support material is removed by vapor etching using hydrochloric acid and the sample is cleaned by immersion in a mixture of 90 % ethanol and 10 % water. As a result, most of the titanium dioxide bridges are retained and can be characterized (see Figure 1). The samples produced still have cracks and spalling on the bridges, but it can be demonstrated that the ATLANT 3D Nanosystems atomic layer 3D printing process can be used for the direct deposition of free-standing three-dimensional structures that can be further used to fabricate components in microelectronics.

NS6-5 Al_2O_3 ALD Buffer Layers for Epitaxial Growth of Boron Nitride Beyond the Self-Termination Limit*, **Mateusz Wlazło,*** CBRTP - Research and Development Center of Technology for Industry, Poland*; P. Caban,* Łukasiewicz Research Network - Institute of Microelectronics and Photonics, Poland*; G. Kołodziej ,* CBRTP - Research and Development Center of Technology for Industry, Poland*; P. Michałowski ,* Łukasiewicz Research Network - Institute of Microelectronics and Photonics, Poland

The Metalorganic Chemical Vapor Deposition (MOCVD) is a recognized method for the growth of BN epitaxial layers. The important progress in the understanding of the MOCVD growth was achieved when it was demonstrated that the growth mode could be changed from 3D to self-terminated one under high reactor pressure and increased in V/III ratio. It is known that the main problem in the deposition of BN layers is that in self-terminated mode the total layer thickness does not exceed 2nm.In this presentation structural properties of BN layers grown directly on the Al2O3, or using Al2O3 buffer layers made by ALD were investigated. Application of one or two buffer layers was investigated as well as Al2O3 buffer properties. The measurements techniques used for investigation of the grown films concentrated on AFM however involved SIMS, ATR spectroscopy, XRR, Raman. The motivation behind this work is to show directions towards the increase of the total layer thickness.

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