Monday Afternoon, October 21, 2019

2D Materials

Room A216 - Session 2D+AP+EM+MI+NS+PS+TF-MoA

2D Materials Growth and Fabrication

Moderator: Sarah Haigh, University of Manchester, UK

1:40pm 2D+AP+EM+MI+NS+PS+TF-MoA-1 Two-dimensional Non-layered Indium Sulfide for Electronic and Optical Applications, Jian Zhen Ou, A Jannat, K Xu, RMIT University, Australia

Tetragonal indium sulfide (In2S3) is a n-type semiconductor enabled by a unique ordered vacancy structure, which have interesting electronic and optical properties. However, its non-layered nature results in the challenge to realize its two-dimensional (2D) form. Here, we demonstrate two approaches to synthesize 2D In2S3. In the first approach, we grow 2D In2S3 with the thickness of single unit cell in wafer-scale using liquid metal as the reactant medium. The first principle calculation reveals that the 2D InsS3 has highly dispersive conduction band with low effective electron mass, forming multiple band-like electronic transport channels. The field effect mobility of the material is measured to be ~60 cm2 V-1 s-1 with a high degree of reproducibility. In the second approaches, we synthesize 2D In2S3 with the thickness of a few unit cells using the liquid phase exfoliation of the bulk powder. It is found that there is an ultra-thin layer of 2D hexagonal indium oxide (In2O3) formed during the exfoliation process, hence forming an inherent 2D In2O3/In2S3 heterostructure. The photoluminescent life time is enhanced compared to In2S3 alone and the NO2 gas sensing performance of the heterojunction is assessed under the illumination of visible light at room temperature. Excellent response and recovery kinetics are observed with the NO2 detection of limit of <0.5 ppb. These two representative examples demonstrate that 2D In2S3 can be a suitable candidate for high performance electronic and sensing devices.

2:00pm 2D+AP+EM+MI+NS+PS+TF-MoA-2 Synthesis of High Quality Monolayer Transition Metal Dichalcogenides using Direct Liquid Injection, *Kathleen M. McCreary, E Cobas, A Hanbicki, M Rosenberger, H Chuang, B Jonker,* U.S. Naval Research Laboratory

In recent years, interest in monolayer transition metal dichalcogenides (TMDs) has rapidly increased, spurred by the possibility for integration into a variety of technologies such as photodetection, flexible electronics, and chemical sensing. While fundamental investigations can be performed on exfoliated flakes or chemical vapor deposition synthesized isolated islands, the limited size resulting from these techniques poses a significant barrier forimplementation of TMDs in technological applications. To overcome these obstacles, new synthesis avenues should be explored. Here, we outline a novel technique that utilizes a commercially available Anneal Sys growth chamber equipped with direct liquid injection (DLI) heads for all precursors. The use of liquid, rather than solid precursors, provides fine control of both metal and chalcogen precursors leading to the synthesis of monolayer MoS₂ across cm² areas. Photoluminescence, Raman, XPS, and conductive AFM are used to evaluate DLI grown MoS_2 , and indicate high quality material having low defect density, with metrics comparable to or better than exfoliated and chemical vapor deposition grown MoS₂.

2:20pm 2D+AP+EM+MI+NS+PS+TF-MoA-3 Understanding and Controlling the Growth of 2D Materials with Non-Equilibrium Methods and in situ Diagnostics, David Geohegan, Y Lin, Y Yu, Oak Ridge National Laboratory; C Liu, G Duscher, University of Tennessee Knoxville; A Strasser, University of Texas at Dallas; A Puretzky, Oak Ridge National Laboratory; K Wang, Intel Corporation, USA; M Yoon, C Rouleau, Oak Ridge National Laboratory; S Canulescu, DTU Nanolab, Technical University of Denmark; P Rack, University of Tennessee Knoxville; L Liang, W Zhang, H Cai, Y Gu, G Eres, K INVITED Xigo, Oak Ridge National Laboratory Atomically-thin two-dimensional (2D) materials, including layered 2D transition metal dichalcogenide (TMD) semiconductors and their heterostructures, exhibit remarkable quantum properties that are envisioned for energy-efficient photovoltaics, flexible optoelectronics, catalysis, and quantum information science. However, significant synthesis and processing challenges currently limit the technologic development of these "all-surface" materials, including wafer-scale, bottom-up synthesis of uniform layers of crystalline 2D materials that are comparable in guality to exfoliated flakes of bulk materials. As-synthesized crystals of 2D TMDs display remarkable heterogeneity on both the atomistic level (e.g., vacancies, dopants, and edge terminations) and on the mesoscopic length scale (e.g., misoriented grains, layer orientations, and interactions with substrates and adsorbates) that can strongly influence the structure and electronic properties in 2D materials. This heterogeneity offers a serious challenge for synthesis and processing, yet offers a tremendous opportunity to tailor functionality.

Here we describe several approaches that are being developed for in situ diagnostic analysis and control of synthesis and heterogeneity. In addition to conventional vapor transport techniques, progress in laser-based approaches for 2D synthesis and modification, such as pulsed laser deposition (PLD) and pulsed laser conversion of precursors, are presented that permit control of the growth environment using time-resolved in situ diagnostics. The non-equilibrium advantages of PLD to form alloys and vertical heterojunctions are demonstrated using the tunable kinetic energy and digital nature of the process. Correlated atomic-resolution electron microscopy and atomistic theory are used to understand the size and stoichiometry of the "building blocks" deposited for synthesis and the forces that guide assembly. 2D crystals are grown directly on TEM grids within custom chambers and transmission electron microscopes where the ability to 'see' every atom in these atomically-thin crystals permits a unique opportunity to understand the forces governing their synthesis and functionality. In situ optical spectroscopy techniques are described to characterize the material's evolving structure and properties, offering the opportunity to 'close the loop' between synthesis and optoelectronic functionality of 2D materials and heterostructures.

Research sponsored by the U.S. Dept. of Energy, Office of Science, Basic Energy Sciences, Materials Science and Engineering Div. (synthesis science) and Scientific User Facilities Div. (characterization science).

3:00pm **2D+AP+EM+MI+NS+PS+TF-MoA-5** Area-Selective Atomic Layer Deposition of 2D WS₂ Nanolayers, *Shashank Balasubramanyam*¹, Eindhoven University of Technology, The Netherlands, Noord Brabant; *M Merkx*, Eindhoven University of Technology, The Netherlands; *E Kessels*, Eindhoven University of Technology, The Netherlands, Netherlands; *A Mackus*, Eindhoven University of Technology, The Netherlands, Netherlands; *A Mackus*, Eindhoven University of Technology, The Netherlands, Netherlands; *A Bol*, Eindhoven University of Technology, The Netherlands, Netherlands

With continued downscaling of device dimensions, ultra-thin two dimensional (2D) semiconductors like WS₂ are considered as promising materials for future applications in nanoelectronics. At these nanoscale regimes, device fabrication with precise patterning of critical features is challenging using current top-down processing techniques. In this regard, area-selective atomic layer deposition (AS-ALD) has emerged as a promising candidate for bottom-up processing to address the complexities of nanopatterning. Till date, AS-ALD of metals¹ and dielectrics² have been successfully demonstrated. However, AS-ALD of 2D materials has remained elusive. In this contribution, we demonstrate area-selective deposition of 2D WS₂ nanolayers by using a three-step (ABC-type) plasma-enhanced ALD process.

AS-ALD of WS₂ was achieved by using acetylacetone (Hacac) inhibitor (A), bis(tertbutylimido)-bis(dimethylamido)-tungsten precursor (B), and H₂S plasma (C) pulses. This process resulted in immediate growth on SiO₂ while a significant nucleation delay was observed on Al₂O₃, as determined from *in-situ* spectroscopic ellipsometry (SE) and *ex-situ* X-ray photoelectron spectroscopy (XPS) measurements. The surface chemistry of this selective process was analysed by *in-situ* Fourier transform infrared spectroscopy (FTIR). The analyses revealed that the inhibitor adsorbed on the Al₂O₃ surface, blocking precursor adsorption, while little or no inhibitor adsorption was detected on the SiO₂ surface where WS₂ was readily deposited. Furthermore, the area-selective growth was demonstrated on SiO₂ samples with patterned Al₂O₃ on top. On SiO₂, WS₂ could be deposited with angstrom-level thickness control.

To improve the crystallinity, the AS-ALD WS₂ films were annealed at temperatures within the thermal budget of industrial semiconductor processing (\leq 450°C). The annealed films exhibited sharp Raman peaks, which is a fingerprint of highly crystalline WS₂. Furthermore, Raman line scans over the patterns showed very sharp peak intensity transitions at the SiO₂-Al₂O₃ interface which confirmed that annealing had no impact on selectivity.

To summarize, this work pioneered the combination of two key avenues in atomic-scale processing: area-selective growth and ALD of 2D materials. It is expected that the results of this work will lay the foundation for area-selective ALD of other 2D materials.

¹ R. Chen and S.F. Bent, Adv. Mater. (2006).

¹ TFD James Harper Award Finalist

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² A. Mameli, M.J.M. Merkx, B. Karasulu, F. Roozeboom, W.M.M. Kessels and A.J.M. Mackus, *ACS Nano* (2017).

3:20pm 2D+AP+EM+MI+NS+PS+TF-MoA-6 Growth Behavior of Hexagonal Boron Nitride on Cu-Ni Binary Alloys, *Karthik Sridhara*, Texas A&M University; *J Wollmershauser*, U.S. Naval Research Laboratory; *L Nyakiti*, Texas A&M University; *B Feigelson*, U.S. Naval Research Laboratory

Controlled growth of large area n-layered chemical vapor deposited (CVD) hexagonal boron nitride (h-BN) is of great interest as a tunnel dielectric, and substrate for graphene and transition metal dichalcogenides (TMDs). The CVD growth of h-BN has been demonstrated on various transition metal catalytic substrates such as Cu, Ni, Pt and Fe. Of these metal substrates, Cu and Ni are frequently used due to their relative abundance and low cost. However, h-BN growth on Cu leads to monolayer films, and growth on Ni yields thicker, substrate grain-dependent films. Therefore, a cost-effective transition metal substrate is needed that will facilitate controlled n-layered h-BN growth.

In this work, we prepare isomorphous Cu-Ni binary alloys from 10-90 wt.% Ni by creating Ni-rich (Ni-Cu) and Cu-rich (Cu-Ni) alloys using electroplating of Cu on Ni foils and Ni on Cu foils, respectively. The electroplated foils are then annealed at ~1030° C for >5 hours to create Ni-Cu and Cu-Ni alloys. The alloys are subsequently polished mechanically to create a planarized surface suitable for h-BN growth. The surface morphology before and after polishing is assessed using a scanning electron microscope (SEM). Energy dispersive spectroscopy (EDS) characterization of the alloys confirms a designed stoichiometry at every weight percent. h-BN is grown on the alloys using atmospheric pressure chemical vapor deposition (APCVD) at 1030° C, with ammonia borane as the precursor, and H_2/N_2 as the carrier gas flowing at ~200 sccm. Cu and Ni foils are used as control samples for this study. Fourier transform infrared reflection absorption spectroscopy (FT-IRRAS) is used to confirm and characterize h-BN growth directly on Cu, Ni and alloy substrates. SEM is performed to evaluate the h-BN film and crystal morphology. The results indicate that the h-BN growth behavior on Ni-Cu is different than on Cu-Ni alloys. A trend of decreasing h-BN amount with reducing Ni concentration is observed on Ni-Cu alloys while no such trend is observed on Cu-Ni alloys. Additionally, there are large (~20 $\mu m)$ multilayer and monolayer single crystals of h-BN on Ni-Cu alloys, and predominantly monolayer crystals and films of h-BN on Cu-Ni alloys. The difference in growth behavior is studied using x-ray photoelectron spectroscopy (XPS) and electron backscattering diffraction (EBSD), which reveal that the alloy surface composition determines the h-BN growth. This work demonstrates how Cu-Ni alloy substrate of different compositions, along with CVD growth conditions, can be used to control h-BN growth.

4:00pm **2D+AP+EM+MI+NS+PS+TF-MOA-8** Chemical Deposition of Vanadium Disulfide on Silicon for Optoelectronic Applications, Mathias Fraccaroli, *R Gassilloud, S Cadot,* CEA-LETI, France; *B Pelissier,* LTM, Univ. Grenoble Alpes, CNRS, France; *C Vallée,* LTM, Univ. Grenoble Alpes, CEA-LETI, France; *A Sylvestre,* G2Elab, Univ. Grenoble Alpes, France

In recent years, the search for alternative substrates to standard semiconductors (Si, Ge, SiGe, III-V, II-VI, etc.) has intensified. In this context, the transition metal dichalcogenides (TMDs) have recently emerged as candidates for the realization of original devices in a context of diversification of functionality (more than Moore). Indeed, these lamellar materials, structurally similar to graphene, have a great diversity of electrical behaviors, from the semiconductor to the metal, as well as many interesting properties (piezoelectricity and photoluminescence for MoS₂ and WS₂, even ferromagnetism by the addition of a dopant, temperature resistive transition for TaS₂, ...). The interest of the scientific community for this family of materials is growing, mainly for the most famous of them: MoS₂ and WS₂. Among this family, vanadium disulfide (VS₂)remains little studied and the development of a transferable synthesis method to an industrial scale remains a real challenge.

In this context, the development of a method of synthesis by atomic layer deposition could allow to consider future application for this material in microelectronics. In fact, due to its inherent uniformity and conformity, atomic layer deposition (ALD) is currently envisaged as a solution to grow these sulfides on 200/300mm silicon wafers.

This presentation will describe for the first time the process developed for VS_2 synthesis on a 300mm silicon wafer. The different growth mechanisms involved with this film were first analyzed by *quasi-insitu* X-ray Photoelectron Spectroscopy (XPS) without airbreak. Also, the compositions were extracted to assess the growth rate and the incubation time and compared to other standard technics such as X-ray reflectometry. Subsequently, the physico-chemical properties of the film obtained by

different will be presented. A focus on the optoelectronic properties of the film will be presented. Indeed, this film is transparent and conductive, an 8nm film has a transmittance of 78% and a resistivity of 784 μ Ohm.cm.

This work has been partially supported by the program EquipEx IMPACT (ANR-10-EQPX-33)

Keyword: Quasi-insitu XPS, Transition Metal Dichalcogenides, CVD.

4:20pm **2D+AP+EM+MI+NS+PS+TF-MOA-9 Controlled Growth of Transition Metal Dichalcogenide Monolayers for Applications in Nanoelectronic and Nanophotonic Devices**, *A George, C Neumann, D Kaiser, R Mupparapu,* Friedrich Schiller University Jena, Germany; *U Hübner,* Leibniz Institute of Photonic Technology, Jena, Germany; Z Tang, A *Winter, I Staude,* **Andrey Turchanin**, Friedrich Schiller University Jena, Germany

Controlling the flow rate of precursors is highly essential for the growth of high quality monolayer crystals of transition metal dichalcogenides (TMDs) by chemical vapor deposition. Thus, introduction of an excess quantity of precursors affects the reproducibility of the growth process and results in the multilayer growth. Here, we demonstrate the use of Knudsen-type effusion cells for controlled delivery of sulfur precursor for the large area, high density, size-controlled and highly reproducible growth of monolayer TMD crystals [1]. The size of the grown crystals can be tuned between 10 -200 μ m. We grow MoS₂, WS₂, MoSe₂ and WSe₂ monolayer crystals as well as MoSe₂-WSe₂ lateral heterostructures and characterize them by optical microscopy, atomic force microscopy, Raman spectroscopy, photoluminescence spectroscopy and electrical transport measurements. It has been found that they possess a high crystalline, optical and electrical quality based on their single crystalline nature. We demonstrate their implementation in novel field-effect and nanophotonic devices and discusse an influence of the point defect density on their functional characteristics [2-3]. Moreover, we present a novel synthetic route for the integration of TMDs into lateral heterostructures with other 2D materials [4].

[1] A. George et al., J. Phys.: Mater. 2 (2019) 016001.

[2] T. Bucher et al., ACS Photonics 6 (2019) 1002.

[3] R. Meyer et al., ACS Photonics 6 (2019) DOI: 10.1021/acsphotonics.8b01716

[4] A. Winter et al., Carbon 128 (2018)106.

4:40pm 2D+AP+EM+MI+NS+PS+TF-MoA-10 Atomic Layer Deposition of BN as a Novel Capping Barrier for B2O3, Aparna Pilli, J Jones, J Kelber, University of North Texas; A LaVoie, F Pasquale, Lam Research Corporation The deposition of boron oxide (B₂O₃) films on Si and SiO₂ substrates by atomic layer deposition (ALD) is of growing interest in microelectronics for shallow doping of high aspect ratio transistor structures. B2O3, however, forms volatile boric acid (H₃BO₃) upon ambient exposure, requiring a passivation barrier, for which BN was investigated as a possible candidate. Here, we demonstrate, deposition of BN by sequential BCI /NH reactions at 600 K on two different oxidized boron substrates: (a) B O deposited using BCI /H O ALD on Si at 300 K ("B O /Si"); and (b) a boron-silicon oxide formed by sequential BCI /O reactions at 650 K on SiO followed by annealing to 1000 K ("B-Si-oxide"). X-ray photoelectron spectroscopy (XPS) data demonstrate layer-by-layer growth of BN on B_2O_3/Si with an average growth rate of ~1.4 Å/cycle, accompanied by some B₂O₃ removal during the first BN cycle. In contrast, continuous BN growth was observed on B-Sioxide without any reaction with the substrate. XPS data also indicate that the oxide/nitride heterostructures are stable upon annealing in ultrahigh vacuum to >1000 K. XPS data, after the exposure of these heterostructures to ambient, indicate a small amount of BN oxidation at the surface NH species, with no observable hydroxylation of the underlying oxide films. These results demonstrate that BN films, as thin as 13 Å, are potential candidates for passivating boron oxide films prepared for shallow doping applications.

5:00pm **2D+AP+EM+MI+NS+PS+TF-MoA-11** Atomic Layer Deposition of **SiO₂ on Group VIII Metals: Towards Formation of a 2D Dielectric**, *T Suh*, *R Yalisove, James Engstrom*, Cornell University

The atomic layer deposition (ALD) of many metals, particularly Group VIII (now known as Groups 8, 9 and 10), on SiO₂ has been an active area of research in many fields, which include microelectronics and heterogeneous catalysis. There have been many fewer studies of the inverse—the deposition of SiO₂ on many of these same metals. One possible reason to explore the ALD growth of SiO₂ on transition metals is that it might provide a route to an atomically thick SiO₂ dielectric, *silicatene*. Silicatene is a 2D

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material that consists of a bilayer of Si_2O_3 linked to each other by bridging oxygen atoms (giving SiO₂), where there are no dangling bonds or covalent bonds to the underlying substrate on which it is grown. For example, an established route to form silicatene involves deposition of elemental Si in UHV and subsequent high-temperature annealing on various singlecrystalline metal surfaces including, but not limited to, Ru(0001), Pt(111), and Pd(100). Such a process, unfortunately, is likely not compatible with high-volume manufacturing. With this motivation we embarked on a study of the plasma-assisted ALD of SiO₂ on e-beam deposited polycrystalline thin films of Ru, Pt and Pd using a commercial ALD reactor. We analyzed both the thin films and the starting substrates using a combination of techniques including contact angle, spectroscopic ellipsometry (SE) and X-ray photoelectron spectroscopy. Thin films of SiO_2 were deposited using tris(dimethylamido)silane and an oxygen plasma at a substrate temperature of 200 °C, and we examined growth for 5, 10, 20, 50 and 100 cycles. Contact angle measurements showed immediate evidence for SiO₂ deposition on all metal surfaces, and the contact angle decreased and remained constant and < 10° from 5 to 100 cycles of ALD. From SE we found little evidence of an incubation period, and growth was linear for the range of sample examined and the thickness deposited per cycle was remarkably constant at a value of 0.76-0.78 Å-cycle⁻¹. Analysis of these films using angle-resolved XPS was consistent with the formation of a thin film of SiO2 with uniform thickness. Having characterized the thin film thickness-ALD cycle relationship we subjected SiO₂ thin films with thickness of ~ 7-15 Å to post-deposition high-temperature anneals in oxygen furnace. Initial attempts to form silicatene with an anneal at 800 °C, produced a structure suggesting possible interfacial reaction between the SiO₂ and Ru, perhaps involving silicide formation. We will end our presentation with a discussion of recent work involving a more extensive examination of the post-deposition annealing step, and deposition on patterned wafers.

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