Wednesday Afternoon, August 1, 2018

Emerging Materials Room 107-109 - Session EM-WeA

MLD & Emerging Materials

Moderators: Jiyoung Kim, University of Texas at Dallas, Charles Dezelah, EMD Performance Materials

1:30pm EM-WeA-1 Physical, Chemical, and Electrical Properties of Molecular Layer Deposited Alucone Thin Films using Trimethyl-aluminum and Hydroquinone, *Seung-Hwan Lee*, *G Beak*, *J Lee*, *J Park*, Hanyang University, Republic of Korea

Molecular layer deposition has been rapidly emerged to fabricate an organic film, showing a self-limited reaction and half-reaction cycle by using metal precursor and/or organic monomer. The deposited films exhibit excellent conformality, uniformity and exact thickness control like atomic layer deposition. Many researcher groups reported alucone films using various organic monomers (ethylene glycol (EG), diethylene glycol (DEG), hydroquinone (HQ) etc.) but they have researched basic process conditions and film properties. [1-2] However, mostly the organic layers are very sensitive to air ambient. The water and oxygen may react with organic films. So, it is one of big challenges to utilize the functional films in emerging application areas.

In this work, we investigated the physical, chemical, and electrical properties of Alucone films, deposited by trimethyl aluminum (TMA) and HQ. The pristine alucone film didn't show any D and G peak in Raman spectra, indicating a carbon domain evidence. But the annealed alucone film did carbon domains like amorphous graphite carbon layers. The pristine films, including carbon-oxygen and carbon-hydrogen bond, dramatically turned to the dehydrated alucon filme during the annealing process. This change may stabilize the organic structure and improve the air-stability. Spectroscopic ellipsometer (SE), Auger electron spectroscopy (AES), Raman spectra and X-ray photoelectron spectroscopy (XPS) were used to understand film properties. Also, hall measurement and seebeck coefficient measurement were used to measure electrical properties of the annealed alucone film, which showed p-type carrier and conductivity. Thus, the process and material will be a promising layer because the stable organic layer may provide not only new functional surface but also functional coted films in emerging electronic applications.

[1] Steven M. George et al, Growth and properties of hybrid organicinorganic metalcone films using molecular layer deposition techniques, Advanced Functional Materials, (2013), 23, 532

[2] Xueliang Sun et al, Safe and durable high-temperature lithium-sulfur batteries via molecular layer deposited coating, Nano letters, (2016), 16, 3545

1:45pm EM-WeA-2 Molecular Layer Deposition of Boron Carbide Thin Films, *Suhaib Malik*, *R Thapa*, *L Dorsett*, *S Wagner*, *A Caruso*, University of Missouri-Kansas City; *D Merrill*, *J Bielefeld*, *S King*, Intel Corp.; *M Paquette*, University of Missouri-Kansas City

The drive towards smaller and more powerful integrated circuits (IC) has put increasing strain on both traditional circuit materials and electronics manufacturing. New materials must perform as better electrical components than their predecessors and, as is increasingly becoming the case, meet higher standards of thermal and mechanical properties for performance and miniaturization. The state of the art approach to building IC components is top-down; a pre-built material of a given thickness is etched into. The advent of atomic layer deposition (ALD) extends a topdown approach by allowing for smarter-designed materials with more exact layering and patterning, while also enabling a bottom-up approach by opening the door to capabilities like selective deposition. Boron carbide (BC) films provide a unique solution to the first half of the above problem: they display promising electrical and physical properties for applications ranging from corrosion protection to low-k dielectrics to electrical surface modification to neutron detection. We propose that if BC can be deposited using an ALD growth scheme, it could address many present challenges in engineering micro- and nano-scale electronics and introduce a unique set of materials to the ALD family for other applications. In this presentation we will describe the use of carboranes as molecular layer deposition precursors in growing thin BC films and the effect of growth parameters on deposition behavior.

2:00pm EM-WeA-3 Achieving Room Temperature and Below Phase Transitions in ALD Doped VO₂ Films, Virginia Wheeler, U.S. Naval Research Laboratory; A Kozen, The American Society for Engineering Education; M Currie, B Downey, D Meyer, C Eddy, Jr., U.S. Naval Research Laboratory

VO₂ undergoes a first order crystalline phase transition at a critical temperature ($T_c = 68^{\circ}C$), resulting in significant changes in intrinsic electrical and optical properties, especially in the infrared. Optical changes with this phase transition are of particular interest as passive and active components of optoelectronic devices, specifically for thermal regulation. Realizing this type of device often requires the integration of thin, conformal VO₂ films with complex, non-planar structures (like metamaterials). Thus, atomic layer deposition (ALD) is the ideal deposition method in these cases. While the T_c of VO₂ may be appropriate for some applications, others require this transition to occur at lower temperatures. Traditionally, lowering the T_c has been accomplished through inducing strain or doping VO₂ films with small amounts (1-5 at%) of transition MQ₂ films. Here, we achieve a T_c less than room temperature through ALD Nb doped VO₂ films.

Previous studies have shown that T_c near room temperature can be obtained through W doping. Unfortunately, the growth window for W(CO)₆ and ozone was above 200°C, which is higher than the vanadium precursor (TEMAV) decomposition temperature, rendering it incompatible with the VO₂ process. A variety of other compatible dopants were explored and Nb had the most potential, allowing the T_c to be reduced with minimal degradation in morphology, structure, and optical transition properties.

ALD Nb-doped VO₂ films were formed using supercycles of Nb₂O₅ (TBTDEA-Nb(V), O₃) and VO₂ (TEMAV, O₃) at 150°C on c-sapphire substrates. By varying the ratio of Nb₂O₅ to VO₂ cycles, doped films with 1-7 at% Nb were achieved. Initial studies suggest that the Nb_2O_5 layer may inhibit the subsequent nucleation of VO₂ layers, resulting in non-linear incorporation of Nb. A reduction in T_c of -11.2°C/Nb at% was attained, which is larger than reports of Nb-doped VO₂ films by other growth methods. Additionally, up to ~1.6 at% Nb, the magnitude of the optical transition is maintained while shifting the T_c to 36°C. To attain a room temperature T_c requires 3.3 at% Nb, which is accompanied by a reduction in optical transmission modulation to ~10%. Similarly, a 5°C T_c was obtained with 5.1 at% but with only a 5% optical modulation. TEM images show that films with a reduced optical modulation still have inclusion of amorphous grains, even after exsitu annealing, which increases with increasing Nb doping. The extent of being able to fully crystallize VO2 films with high Nb contents will be discussed to determine the realization of usable films with Tc near 0°C.

2:15pm EM-WeA-4 Ozone Based High Temperature Atomic Layer Deposition of SiO₂ Thin Films, *Su Min Hwang*, *X Meng*, *A Lucero*, *H Kim*, *S Kim*, The University of Texas at Dallas; *B Hwang*, Dow Chemicals; *J Kim*, The University of Texas at Dallas

Recently, deposition of SiO₂ films with high quality and good conformality has drawn great attention as device structures have become more complicated. Conventionally used LPCVD and PECVD for SiO₂ deposition have limitations in terms of conformal deposition and thickness scalability. Since ALD is expected to overcome the challenging issues, several research groups have reported SiO₂ ALD using SiCl₄, SiH₂Cl₂, and Si₂Cl₆ at low temperature (250 – 500°C)). Unfortunately, the ALD films show less dense film qualities compared to thermal oxide [1-3]. For high quality SiO₂ films with good conformality, an ALD process at high temperature is desirable. However, research on high temperature ALD processes is scarce due to the difficulty of the process caused by decomposition of precursor resulting in CVD-like growth instead of self-limiting ALD growth.

In this work, oxidation of silicon wafers using various oxidants (H₂O, O₂ and O₃) was performed using a rapid thermal ALD system to identify if O₃ has higher reactivity for high temperature ALD of SiO₂. Although O₃ has a short lifetime above 300°C due to thermal decomposition, thermal oxide grown with O₃ at 300 to 800°C shows higher thickness compared to oxide films grown using H₂O and O₂. The activation energy of O₃ based process was calculated to 0.07 eV, which is a lower value than those of H₂O based process (0.14 eV) and O₂ process (0.16 eV). Among the possible oxidants, we confirmed that O₃ has a higher reactivity for ALD of SiO₂ due to reactive O radicals. Based on these results, ALD of SiO₂ thin films using Si₂Cl₆ and O₃ was performed at from 500 to 700°C. At 650°C we observe a saturated growth rate of 0.24 Å/cycle at a Si₂Cl₆ exposure of 2.0 10⁶ L with fixed O₃ exposure. Growth rate is linear with the number of cycles indicating there is only a small or no incubation period observed. Our findings highlighted

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that reactivity of O3 can be maintained at high temperature and it can be applicable to ALD of SiO₂ process.

We acknowledge TMEIC for ozone generator.

2:30pm EM-WeA-5 Atomic Layer Deposition of High-Tc Magnesium Diboride (MgB₂) Film for Superconducting Radio-Frequency Particle Beam Accelerators, Alireza Nassiri, D Mandia, A Yanguas-Gil, A Mane, J Elam, Argonne National Laboratory

Atomic Layer Deposition (ALD) as a self-limiting coating process is an ideal technique relatively thick, high quality coating of high-Tc superconductor Magnesium Diboride (MgB₂) on radio frequency copper resonant structures (cavities) for particle beams accelerators. MgB2 is considered one of the viable materials to substitute bulk niobium for superconducting radio frequency cavities. Utilizing a MgB2 coating on the inner wall of a copper cavity will allow operation at higher temperatures (20-25 K) than Nb cavities due to the high transition temperature of MgB₂ (39 K) and the high thermal conductivity of Cu. Achieving higher accelerating fields and eliminating expensive capital and operating costs of large liquid helium refrigeration system by using highly efficient and cost effective cryo-coolers are amongst the major benefits of using magnesium diboride. Here, we will present initial results on this ongoing effort.

2:45pm EM-WeA-6 Commerical Production of ALD-Coated Powders and Polymers, Daniel Higgs, ALD NanoSolutions, Inc.

Atomic and molecular layer deposition (ALD) can be economically scaled to coat tons of powder, kilometers of polymers and many thousands of specialty objects a day. ALD reached commercial scale for coating various chemistries onto silicon wafers in the semiconductor industry in the 1990s, which today is a multi-billion dollar market. ALD is now becoming the method of choice for coating ultrathin, precisely controlled films onto particles and other new material substrates.

This talk will discuss the cost-effective commercial production of ALDenabled materials at >1000T/yr of powders and >50M m2 /yr of polymers. We will cover batch and continuous ALD systems that enable this high volume production as well as discuss various markets for the materials processed in these systems. Two current commercial products will be touched on and other pre-commercial products will be discussed.

ALD Nano helps customers solve particle material challenges by perfecting the necessary ALD coating chemistries, process conditions and manufacturing equipment systems. Our global leadership position is ALD on powders, also known as particles or particle ALD. Many Fortune 500 companies are among our customers and supply channel partners. ALD on particles has many benefits including:

• Superior precision and cost-effectiveness compared with chemical vapor deposition (CVD) and other techniques enable new or better applications for known substrates with improved end-device performance, lifetime, cost and safety.

• Novel atomic-scale material designs can be created with significant value to materials supply chain customers and end-device partners.

· Substrate functionality improvements occur by changing physical characteristics of particles including enhanced (or reduced) chemical stability, flowability, corrosion, oxidation, conductivity, hydrophobicity, optical properties, material compatibility, dispersion, sintering control, barrier performance, and other improvements that result in better end device performance.

· Particle agglomeration does not happen with proper ALD process conditions, regardless of particle size, shape and morphology.

3:00pm EM-WeA-7 Ruthenium: Advanced Nodes and Supply Chain Implications, Oliver Briel, D Zeng, A Wilk, Umicore AG & Co. KG, Germany Advanced new materials and chemistries may play an important role to extend Moore's law to continue miniaturization of chip design while at the same time increasing their performance. One of these promising materials is ruthenium partly due to its unique properties of interest to the industry.

In the early 2000's, there was a Ru hype at the R&D scale, however little or none of the new materials made to HVM, because the industry solved the problems by extending existing technologies, or by introducing alternative materials. Today we are in a similar situation as then except it seems to be more difficult to extend lifetime of the existing technologies or find other alternatives, hence we sense the optimism in ruthenium adoption for advanced chip nodes as foreseen in technology roadmaps provided by leading research organizations supporting the industry. While the outcome of Ru adoption remains to be seen, one thing we have learned is that many industry participants shared reservation in ruthenium based application Wednesday Afternoon, August 1, 2018

development, because of the notion this material system being very "expensive" with "unpredictable price movements" and "unstable supply".

Umicore is a traditional platinum group metals company, active in the field for many decades, if not centuries. Through this presentation and discussion, we hope to shed light on and demystify the black box of PGM market dynamics. We will discuss a number of topics, such as where do PGMs come from, how much are available, which technologies/applications are the drivers of their demands, how are the PGMs priced, what procurement strategies can be used to mitigate cost impact due to price fluctuations, how does metal reclaim work, and who are among the key players in the value chain, all with a special focus on ruthenium.

In addition, we will introduce our most recent advanced Ru metal precursors as potential candidates for ALD and MOCVD applications.

3:15pm EM-WeA-8 An Alternative Precursor for Safe Deposition of Aluminum Oxide Thin Films, Liao Cao, Ghent University, Belgium; F Minaye Hashemi, Delft University of Technology, Netherlands; F Mattelaer, Ghent University, Belgium; J van Ommen, Delft University of Technology, Netherlands; C Detavernier, Ghent University, Belgium

Aluminum oxide is widely used as a barrier layer, dielectric film and encapsulation material due to its excellent chemical and thermal stability, high field strength and high resistivity. Controlled deposition of aluminum oxide thin films via atomic layer deposition (ALD) is a well-developed process with applications ranging from semiconductor electronics to largescale coatings. The most commonly used precursor for deposition of Al₂O₃ via a thermal ALD process is trimethylaluminum (TMA). TMA has a high vapor pressure and is very reactive towards most surfaces, acting as an ideal precursor for deposition of Al₂O₃ at various temperatures. However, TMA is pyrophoric, toxic, corrosive and expensive when aiming to coat surfaces at large scale, such as coating powders or roll-to-roll processes. Thus, alternative low-cost and safe precursors for deposition of alumina would facilitate economical and environmentally sustainable manufacturing of new surfaces and materials.

In this work we investigate an alkoxide precursor as a safe and stable alternative to TMA. Aluminum tri-isopropoxide (TIPA) is studied as the Al source for ALD of Al₂O₃ when different oxidizing agents including water, plasma and ozone are employed in the deposition process. We have explored the deposition of Al₂O₃ using TIPA in ALD systems operating in vacuum and atmospheric pressure conditions. Deposition process has been studied on both planar substrates and on different nanoparticles to explore the feasibility of using TIPA for various purposes including large-scale applications.

During thermal and plasma processes in vacuum ALD, we show a growth window of Al₂O₃ from 140°C up to 300°C. Growth rates achieved in the plasma process are comparable to the thermal ALD of TMA and water (1.1Å/cycle). X-ray reflectivity analysis confirm an increasing density of Al₂O₃ film when deposition temperature is increased. Scanning electron microscopy and atomic force microscopy show deposition of smooth films when TIPA is used as a precursor. X-ray photoelectron spectroscopy studies confirm stoichiometric deposition of Al₂O₃ with no presence of carbon contamination. The thermal water process at atmospheric pressure ALD (AP-ALD) resulted in a growth rate up to 1.1 Å/cycle with no residual carbon. No growth was observed when ozone was used as the oxidizing agent under atmospheric pressure conditions. AP-ALD on nanoparticles shows different growth modes on TiO₂ versus SiO₂ nanoparticle surfaces confirmed by transmission electron microscopy analysis. Using TIPA as an ALD precursor would open up the possibility for a safer and cost-effective process for deposition of Al₂O₃ in various applications.

4:00pm EM-WeA-11 Application of PEALD Technique to the Fabrication of Vertical TFT for the Ultra High-Resolution Display, Kwang-Heum Lee, S Lee, H Yeom, J Ko, Korea Advanced Institute of Science and Technology, Republic of Korea; C Hwana, Electronics and Telecommunications Research Institute; S Park, Korea Advanced Institute of Science and Technology, **Republic of Korea**

1. Introduction

In nowadays, VR (Virtual Reality) and AR (Augmented Reality) are thought to be the key applications of future display. However, there still remain several obstacles, and one of them is the low resolution of display. To solve this problem, higher pixel density of several thousand PPIs is strongly required. Oxide semiconductor vertical thin-film transistor (VTFT) can be an appropriate candidate for the backplane component of the ultra highresolution display. Its vertical channel structure makes it possible to reduce

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footprint size, which helps to make the pixels to be smaller and brighter at the same time. And also, it is possible to fabricate sub-micron scale channel length TFT by using the conventional photolithography techniques for the display industry more easier. This can give us high on-current characteristics. With these reasons, we conducted research for the oxide semiconductor based VTFT and tried to improve its electrical properties.

2. Experiments

Oxide semiconductor based VTFTs were fabricated on thermally oxidized Si wafer substrates. We used ITO for the source/drain electrodes, SiO₂ for the spacer, InO_x via plasma-enhanced atomic layer deposition (PEALD) method for the active layer, and Al₂O₃ for the protective layer (PL) + gate insulator. PL layer was adopted to reduce gate leakage current. All patterning steps except for the active and PL layer were conducted by dry-etching. Although, VTFTs have vertical spacer with taper angle of ≈80°, it does not make any problems with step coverage due to adopting PEALD technique for deposition of active & gate insulator layers.

3. Results

From our experiments, we could fabricate the VTFT with high on-current driven at low voltage. In addition, through pre-annealing process of InO_x active layer under oxygen atmosphere, we could verify broader process window for the post-annealing, which is needed for the LCD display. These results indicates the possibilities of VTFT for the Ultra High-Resolution display.

4. Conclusions

In this paper, we investigated the possibility of fabricating oxide semiconductor VTFT using PEALD. Through this result, we will continue to conduct our experiments for the process optimization and adopting to the ultra high-resolution display panel.

4:15pm EM-WeA-12 Effect of Substrate on MoS₂ Deposited by Plasmaenhanced Atomic Layer Deposition, *Asad Mughal*, *T Walter, K Cooley,* The Pennsylvania State University; *A Bertuch,* Veeco-CNT; *S Mohney,* The Pennsylvania State University

Due to their promising physical properties, semiconducting twodimensional transition metal dichalcogenides (2D TMDs) are a materials class receiving intense research attention. Molybdenum disulfide is one of the most widely studied materials in this class and has the potential for applications ranging from electronics to catalysis. However, achieving large-area uniform growth of MoS2 is challenging and is typically accomplished at elevated temperatures using chemical vapor transport, chemical vapor deposition, or related techniques. Plasma-enhanced atomic layer deposition (PEALD) is a promising method for achieving control of film thickness at the atomic scale and with growth temperatures low enough to accommodate integration into traditional semiconductor device fabrication processes. In this work, we use PEALD to grow thin layers of MoS₂. Using the co-reactants (N^tBu)₂(Nme₂)₂Mo and H₂S:Ar plasma, depositions were carried out in a hot-walled PEALD system from 250-450 °C. Higher purity layers were achieved using a sapphire rather than silica plasma tube. Films were characterized by spectroscopic ellipsometry, XPS, TEM, AFM, and resonance Raman spectroscopy (RRS) to determine growth rate, morphology, purity, and crystalline quality. RSS revealed the number of layers of MoS₂ ($A_{1g}(\Gamma)$ - $E_{2g}(\Gamma)$) as well as crystalline quality ($A_{1g}(\Gamma)/LA(M)$). Interesting variations were observed among films deposited on different substrates, with better crystalline quality on (0001) sapphire than on SiO₂/Si or (0001) GaN/sapphire. Molybdenum disulfide was controllably grown down to single-layer thicknesses by adjusting the number of growth cycles. When deposited on silicon nitride membranes, MoS₂ layers were found to be polycrystalline with the (0001) plane parallel to the substrate. However, substrate-dependent out-of-plane growth was observed for thicker films, especially on silicon nitride, SiO2/Si, and sapphire. Higher growth temperatures, as well as post-deposition annealing under sulfur vapor, was shown to increase the crystallinity of the films. We anticipate that this PEALD growth technique will be suitable for a wide array 2D TMDs thin films and devices.

4:30pm EM-WeA-13 Single-crystal Ternary Perovskite YAlO₃ Epitaxial Growth on GaAs and GaN via Y_2O_3 Template Overcoming a Large Film/Substrate Lattice Mismatch, *L. Bo-Yu Young*, *C Cheng*, *K Lin*, *Y Lin*, *H Wan*, National Taiwan University, Republic of China; *R Cai*, *S Lo*, Industrial Technology Research Institute, Republic of China; *M Li*, National Applied Research Laboratories, Republic of China; *C Hsu*, National Synchrotron Radiation Research Center, Republic of China; *J Kwo*, National Tsing Hua University, Republic of China; *M Hong*, National Taiwan University, Republic of China

It is a challenge to perfect a hetero-epitaxial growth. Earlier successful examples are rare-earth metals/Nb/sapphire,¹ which led to the discovery of anti-ferromagnetic coupling through non-magnetic media, based on which the giant magnetoresistance (GMR) for the high-density recording was established, and GaN/sapphire, which led to the blue light emitting diode and lasers. Integrating atomic layer deposited (ALD) single-crystal perovskite onto semiconductor combines rich properties of the perovskites with advanced electronic and opto-electronic devices. Previously we presented hexagonal perovskite YAlO₃ (H-YAP) on GaAs and GaN. We have achieved excellent single crystal H-YAP on GaAs(111)A using ALD sub-nano-laminated Y₂O₃/Al₂O₃ multi-layers through post-deposition rapid thermal annealing with temperatures above 900°C.^{2,3}

From the high-resolution scanning transmission electron microscopy (STEM) images, we have observed a single atomic layer of Y-O at the YAP/GaAs(001) and /(111)A interfaces. In this work, we have further investigated the initial growth of the H-YAP on GaAs(111)A and GaN with and without a Y2O3(111) single crystal template. Fig. 1 shows the X-ray diffraction results of H-YAP grown on the Y₂O₃ template after 900°C anneal. The pronounced fringes shown in Fig. 1(a) indicate excellent crystallinity of H-YAP and sharp interface between H-YAP and Y₂O₃. The narrow FWHM ~0.019° of H-YAP(0004) θ-rocking scan confirmed the excellent crystallinity. From the STEM image shown in Fig. 2, H-YAP grew directly from Y₂O₃. The Y-sub-lattice in the atomic packing of Y2O3(111) has a hexagonal-like structure with a 2-D lattice constant a= 3.75 Å, similar to that of H-YAP(0001) with a = 3.68 Å as shown in Fig. 3. The top Y (Y-O) layer in the Y₂O₃(111) is the initial Y (Y-O) layer of the H-YAP. Notice their structural similarity. The excellent crystallinity of H-YAP may be attributed from the excellent starting Y2O3 layer with a FWHM ~0.017° of Y2O3(444) θ-rocking scan. For the H-YAP/GaAs(111)A, the FWHM of H-YAP(0004) θ-rocking scan is ~0.026° despite a large lattice mismatch ~8.5%. Fig. 4 shows the STEM image of the H-YAP grown on GaAs(111)A, where a Y-O layer was observed between H-YAP and GaAs dumbbell at the interface. This Y-O layer may also be the starting layer of the single crystal H-YAP. Our recent work on the initial growth of ALD- Y₂O₃ on GaAs using in-situ synchrotron radiation photoemission may shed the light on the understanding of the interfacial electronic characteristics, namely the chemical bonding.⁴

LBY, CKC, and KYL have contributed equally to this work.

CHH, JK, and MH are the corresponding authors.

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