Tuesday Morning, December 13, 2016

Thin Films Room Makai - Session TF-TuM

Nanostructured Surfaces & Thin Films II Moderator: Sumit Agarwal, Colorado School of Mines, USA

8:00am TF-TuM1 Ways to Increase the Strength of Langmuir Monolayers of Particles at Air/aqueous Interfaces, *Cathy McNamee*, Shinshu University, Japan; *S. Fujii*, Osaka Institute of Technology, Japan; *S. Yusa*, University of Hyogo, Japan; *M. Kappl*, MPIP, Germany

The use of Langmuir monolayers of polymer particles stabilized at air/liquid surfaces in medical and industrial applications are affected by their physical properties, e.g., stiffness (deformability), their resistance against breakage upon impact of other materials, and their adhesion to other surfaces. We previously studied the physical properties of a Langmuir monolayer of polystyrene particles loaded with poly(N,N-dimethylaminoethyl methacrylate) (particle abbreviation: "PDMA PS") at air/aqueous interfaces by using the Monolayer Particle Interaction Apparatus [1]. A particle was attached to an Atomic Force Microscope cantilever (probe), which acted as the colliding material. The probe was brought from the bulk water to the monolayer of PDMA PS particles at the air/water interface and then returned back into the bulk water, during which time the forces between the probe and the monolayer were measured. A monolayer of PDMA PS particles at the air/water interface was seen from the forcedistance curves to give a low stiffness, a result explained by the induced movement of the particles in the monolayer at the air/water interface by the probe, when the probe was brought into contact with the monolayer from the bulk water.

In this study, we aimed to create a Langmuir monolayer of polymer particles at an air/water interface that showed a high stiffness, even after the collision of a probe or another material. This was achieved by adding a poly(2-hydroxyethyl methacrylate) ("PHEMA") polymer to a Langmuir monolayer of PDMA_PS particles at an air/water interface. The mixed PHEMA- PDMA_PS monolayer gave a polymer-like monolayer at low surface pressures and a particle-like monolayer at high surface pressures. The PDMA_PS particles formed small aggregates that were dispersed throughout the PHEMA monolayer at low surface pressures, a result suggesting that the particles were trapped in the PHEMA network. The stiffness of the mixed monolayer was independent of the surface pressure, but increased as the ratio of PHEMA in the mixed monolayer increased. The stiffness increase was explained by the PDMA PS particles being embedded in the PHEMA polymer network, which inhibited the movement of the PDMA_PS particles by the probe. A PHEMA polymer monolayer containing a small amount of PDMA_PS particles gave a stiffer film than a pure PHEMA polymer monolayer. It was therefore concluded that the stiffness of a particle monolayer could be changed by adding a polymer to a Langmuir monolayer of particles.

[1] McNamee, C.E.; Fujii, S.; Yusa, S.; Azakami, Y.; Butt, H.-J.; Kappl, M. Colloid Surf. B-Biointerfaces, **2015**, *470*, 322-332.

8:20am **TF-TuM2 Carbon Incorporation into the Si(111)-7x7 Surface**, *E. Seo, D. Eom*, Korea Research Institute of Standards and Science (KRISS), Republic of Korea; *J.-M. Hyun, H. Kim*, Sookmyung Women's University, Republic of Korea; *Ja-Yong Koo*, Korea Research Institute of Standards and Science (KRISS), Republic of Korea

Carbon incorporation into Si wafer has attracted much attention due to the important role of carbon in developing high-performance, Si-based devices. The isovalent C atoms may induce systematic surface strains for building nanostructures on Si wafer without the doping effects. However, the extremely low solubility of C atoms in bulk Si (below 0.001%) has been considered as an obstacle to C incorporation into Si surface.

C atoms can be incorporated into the 4th delta-layer (0.4 nm deep) under the Si(001) surface upto the density of 0.125 monolayer with the c(4x4) surface reconstruction.

We investigated the incorporation of C atoms into Si(111)-7x7 surface by using scanning tunneling microscopy. The C atoms are extracted by thermal dissociation of CO molecules adsorbed on the Si(111)-7x7 surface. Previous studies negate adsorption of CO molecules on this surface even at low temperatures near 10 K. However, we find that every surface dangling bond on the Si(111)-7x7 surface can bind strongly with the C atom of a CO molecule even at temperatures higher than room temperature. Especially

CO molecules bind with Si adatoms in three configurations; one <on-top> and two <back-bond inserted> structures.

By thermal annealing, C atoms are incorporated under the Si adatoms of Si(111)-7x7 surface. The maximum density of incorporated C atoms on Si(111)-7x7 is low compared with that on Si(001)-2x1. At high density, C atoms are swept away from the 7x7 reconstructed surface due to the excessive surface stress, forming irregular SiC phases along the step edge.

Regular and uniform C-incorporated Si(111)-V3xV3 reconstruction is not formed on this Si wafer surface.

8:40am TF-TuM3 Optical, Electronic and Structural Properties of Eu and Gd bis-phthalocyanines Thin Films for Optoelectronics, E. Maresova, *Michal Novotny*, Institute of Physics of the Czech Academy of Sciences, Czech Republic; P. Fitl, J. Vlcek, University of Chemistry and Technology Prague, Czech Republic; M. Vondracek, P. Hubik, L. Fekete, Institute of Physics of the Czech Academy of Sciences, Czech Republic; M. Vrnata, University of Chemistry and Technology Prague, Czech Republic; J. Bulir, Institute of Physics of the Czech Academy of Sciences, Czech Republic; B. Paruzel, J. Pfleger, Institute of Macromolecular Chemistry of the Czech Academy of Sciences, Czech Republic; R. Slota, Faculty of Chemistry, Opole University, Poland; J. Sebera, Institute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pis, Elettra - Sincrotrone Trieste S.C.p.A., Italy; I. Kratochvilova, J. Lancok, Institute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of Sciences, Czech Republic; I. Pisitute of Physics of the Czech Academy of

Phthalocyanines (Pcs) exhibit unique properties, i.e. semiconductivity, photoconductivity, chemical stability and optical absorption in the UV-VIS-NIR region. These properties make the Pcs suitable candidates for optoelectronic devices, small molecular organic solar cells, laser printers or chemiresistive gas sensors. Pcs belong in the most efficient class of organic semiconductors for solid state photovoltaic conversion. Metal 'mono'-Pc compounds, i.e. ZnPcs, CuPcs, are most commonly utilized. Lanthanide bisphthalocyanines (LnPc₂) based on two macrocycles coordinated by the rare earth metal offer additional possibilities since their arrangement allows changing the distribution of electronic density within the cores of the particular phthalocyanine moieties whereas the molecular structure of the sandwich complex remains intact.

Bis-phthalocyanine thin films of GdPc2 and EuPc2 were grown by vacuum evaporation from effusion cell. The optical properties were characterized by spectrophotometry and spectral ellipsometry. The spectra exhibited well pronounced Q, B, N and C bands and a band around 460 nm that could be attributed to radical band (X band) – transition $[2e_g(\pi)-2a_{1u}(\pi)]$ suggesting Ln³⁺Pc₂. Relatively high values of conductivity (1.3·10⁻⁵ S.cm⁻¹ for EuPc₂ and 6.1·10⁻⁵ S.cm⁻¹ for GdPc₂) were obtained in open air at room temperature with interdigital surface Au electrodes arrangement. The conductivity was found to be sensitive to the thermal annealing and ambient conditions. Electronic structure was analyzed by XPS, resonant PES and NEXAFS. Well pronounced individual components of core level spectra of C 1s, N 1s, Eu 4d and Gd 4d and resonating lanthanide 4f levels in valence band were obtained and analyzed. The analyses confirmed Ln³⁺ ionic state. NEXAFS measurement supported by density functional theory calculations revealed rather inclined orientation of LnPc2 molecules on the substrate. The molecular orientation could be manipulated by deposition pressure and depends on the film thickness. Morphology was studied by SEM and AFM. The surface roughness was found to be lower than 1 nm. FTIR characterization suggested low deterioration of LnPc₂ in the films.

The obtained results suggest both $GdPc_2$ and $EuPc_2$ as promising candidates for application in photovoltaics.

9:00am TF-TuM4 Defect-induced lOcalized States on Nitride-Based HEMT and Their Influence on the Radiative Recombination Processes, Manolo Ramírez López, Instituto Politécnico Nacional, Mexico; L. Janicki, R. Kudrawiec, M. Baranowski, J. Misiewicz, Institute of Physics, Wroclaw University of Technology, Poland; M. Zhao, K. Kai Cheng, IMEC, Belgium

Intensive efforts have been made to improve electrical performance of GaN-based HEMT transistors but it has not been identified factors that limit the breakdown voltage, electrical mobility and lead to the early device failure[1]. Incorporation of impurities, point defects, dislocations and grain boundaries formed at semiconductor layers of the device are good candidates to explain their electrical limitations. This work was focused to study the effect of non-intentional carbon concentration on optical recombination processes in AlGaN buffer layer and GaN channel of HEMT transistors grown by MOCVD technique. Samples were grown by changing growth temperature from 950 to 1040 °C, which reduced carbon concentration from 8x10¹⁹ to 5x10¹⁷ atoms/cm³ (measured by SIMS). Low temperature photoluminescence (PL) spectra of AlGaN buffer layers where

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show yellow band(YL), blue band(BL) and near band emissions (NBE), whose relative intensities are well related with dislocation density and carbon incorporation. Optical quenching phenomena on YL, BL and NBE was observed as temperature increases and NBE peak position suffers a redshift as carbon concentration decreases [2]. In samples with high carbon concentration (8x10¹⁹ and 2x10¹⁹ atoms/cm³) only carrier redistribution within localized state was observed, while for samples with lowest carbon concentration (5x10¹⁷ to 3x10¹⁸ atoms/cm³) two optical quenching process were identified (carrier redistribution and delocalization). Carrier delocalization was confirmed by yellow-band intensity increase when this process was thermally activated at 150 K. Results are explained in terms of carbon-generated localized states with different depth and aluminum fluctuations within AlGaN buffer layers. High-resolution PL spectra of Cdoped layers show free A exciton and acceptor bound-exciton whose intensities are correlated with crystal quality and carbon concentration. Complementary analysis will be presented for GaN channel of HEMT transistors.

[1] E. Zanoni, M. Meghini, A. Chini, D. Marcon and G. Meneghesso, IEEE Transactions On Electron Devices, **60**, 10 (2013).

[2] M. A. Reshchikov, D. O. Demchenko, A. Usikov, H. Helava, and Yu. Makarov, Phys. Rev. B **90**, 235203 (2014).

This work was performed within the grant of the National Science Center (NCS) (OPUS 5 No. 2013/09/B/st7/02395), and supported by SIP-IPN project No. 20160610. The authors acknowledge to CONACYT for financial support though postdoctoral scholarship No. 265970.

9:20am TF-TuM5 Comparative Study of the Ni-Silicide Films formed on Si and Strained Si:P, Seongheum Choi, J. Kim, J. Choi, S. Cho, Sungkyunkwan University, Korea, Republic of Korea; M. Lee, E. Ko, Yonsei University, Korea; I. Rho, C.H. Kim, SK Hynix Inc.; D.-H. Ko, Yonsei University, Korea; H.S. Kim, Sungkyunkwan University, Korea, Republic of Korea

The epitaxially-grown strained Si:P film can be used as an uni-axial stressor and/or a low resistance contact material in the n-channel metal-oxidesemiconductor field-effect transistors (NMOSFETs) [1]. Because an additional silicidation process is expected on top of the strained Si:P layer, there have been a few attempts to synthesize metal-silicide films, such as Ti- and Ni-silicides [2, 3].

In this presentation, we will compare the microstructural properties of the NiSi films formed on a Si substrate and a strained Si:P film. The strained Si:P film (P concentration of ~1.9%) was epitaxially grown on Si using a low-pressure chemical vapor deposition system. After HF cleaning and Ni deposition, silicidation was performed by rapid thermal annealing at 400~800 °C for 1 min in N₂ ambient. According to microstructural and electrical comparison with the NiSi film formed on Si, a number of large-sized grains with a flat interface existed and a delay of thermal agglomeration was observed in the NiSi film on Si:P. The possible difference in the grain-growth mechanism of the NiSi films on Si and Si:P will be discussed.

9:40am TF-TuM6 Low Temperature Chemical Vapor Deposition of Manganese and Iron Nitride from Bis(2,2,6,6 Tetramethylpiperidido)metal (ii) and Ammonia, *Elham Mohimi, B.B. Trinh, S. Babar, G.S. Girolami, J.R. Abelson,* University of Illinois at Urbana-Champaign, USA

Manganese and iron nitride phases are an interesting class of materials for applications such as optoelectronics, microelectronics, magnetic recording, and spintronics. Since chemical vapor deposition (CVD) can conformally coat high aspect ratio (deep) features used in design of nanoscale devices, it is highly desirable to develop CVD routes for manganese and iron nitride films. However, the lack of suitable precursors has hindered this development.

Here, we report CVD growth of manganese and iron nitride films from a novel and practical precursor, bis(2,2,6,6 tetramethylpiperidido)metal (II), M (tmp)₂, with ammonia as co-reactant. The manganese and iron tmp complexes can be synthesized in high yield and are thermally stabile at room temperature. Growths are done in a cold wall, high vacuum chamber at substrate temperature of 50-350 °C. Precursors are heated to 60-75°C and delivered to the chamber using Ar carrier gas at flow rates of 5-40 sccm. Anhydrous ammonia is delivered through a separate line to afford a partial pressure of 0-13 mTorr.

XPS reveals a compositional ratio of Mn:N = 2.6:1 to 2.8:1 for manganese nitride (Mn_xN_v) and Fe:N = 4.1:1 for iron nitride (Fe_xN) films grown in the temperature range examined, with no carbon contamination within the detection limits of instrument. All films show a slightly columnar

microstructure in cross-section SEM. While there was a few min of nucleation delay for Mn_xN_y growth at 150 °C, no nucleation delay was observed for Fe_xN under the same growth conditions. There is no film growth at all in the absence of ammonia; this indicates that transamination is required to create Mn-NH₂ groups that react on the substrate surface to form the nitride film, as seen for film growth from other metal amido precursors in the presence of ammonia. Films are conformal in micro-trenches of aspect ratio 3; the thickness profiles are, however, characteristic of film growth from two reactive species, one with high and one with low sticking coefficient at the film surface. Copper diffusion barrier properties of a 12 nm manganese nitride film are investigated by annealing a test structure of 200 nm Cu / 12 nm Mn_xN_y / 300 nm SiO₂ / Si at 500 °C under Ar for 1 hour. Auger electron spectroscopy depth profiles show that manganese nitride performs well as copper diffusion barrier for microelectronic applications.

10:20am **TF-TuM8 Multi-functional Thin Film Coatings formed via Nanoscale Grinding**, *R. Sapkota*, *Chris Papadopoulos*, University of Victoria, Canada

Nanostructured surfaces and thin films composed of nanoscale particles can be created using a variety of nanofabrication methods for applications in electronics, photonics, energy, biotechnology, etc. Generally, such nanostructures can take many forms depending on the various top-down to bottom-up fabrication methodologies [1]. *Nanoscale grinding*, or colloidal grinding, is a unique approach to nanoparticle thin film synthesis that can directly produce large amounts of nanoscale particles in an appropriate solvent without the use of complex chemical or physical processing. Planetary ball milling is typically used to grind a starting bulk powder into a nanoscale colloidal suspension suitable for thin film coating of various functional materials from solution. Efficient energy transfer during planetary ball motion leads to a fast and inexpensive process for the size and dispersion of the particles can be controlled and optimized for applications.

Here we describe results using the nanoscale grinding approach to fabricate multi-functional nanostructured thin film coatings. Nanogrinding allows tunability of film properties that can lead to novel functions depending on the particular combination of material, solvent and nanoparticle geometries/dimensions used. These unique abilities produce a general solution-based processing approach for thin film surface coatings and devices (including non-planar geometries).

Nanostructured thin films based on grinding high-purity powders (Si and TiSi) and characterization via electron microscopy, scanning probe, electronic transport and contact angle measurements are presented. Zirconia beads in a planetary ball mill with readily available powders initially consisting of ~10-50 micron particles in solvent were used. Grinding speeds and times varied between approx. 300-1000 rpm and 10-250 minutes, respectively. The resulting colloidal dispersions are deposited on a substrate for analysis. As grinding time is increased, the particles and films display good uniformity with sizes reaching below 100 nm. Both electrical conductance and contact angle could be tuned over a wide range depending on grinding conditions thereby creating unique multi-functional nanostructured materials and films. We also discuss preliminary results on optical and photocatalytic properties of the nanostructured surfaces. Thin films created via nanogrinding possess unique and varied electrical, optical and mechanical properties, which can be used to create advanced materials and devices.

[1] C. Papadopoulos, "Nanofabrication: Principles and Applications", Springer, 2016.

10:40am **TF-TuM9** Optimization of the ZnO Passivation Process on p-type In_{0.53}Ga_{0.47}As Using Atomic Layer Deposition, *Changmin Lee*, *Y. An, S. Choi, J. Song*, Sungkyunkwan University, Republic of Korea; *Y.-C. Byun, J. Kim*, University of Texas at Dallas, USA; *H.S. Kim*, Sungkyunkwan University, Republic of Korea

For the performance enhancement of the inversion-type III-V metal-oxidesemiconductor field-effect transistors (MOSFETS), it is essential to improve the interface quality between the high-*k* gate dielectric and the p-type III-V substrate. Recently, ZnO passivation using an atomic layer deposition (ALD) process was reported to be effective in removing the interface oxides and improving the electrical properties on both p-type GaAs [1] and p-type $In_{0.53}Ga_{0.47}As$ [2].

In this study, the ALD-ZnO treatment was performed on the sulfurpassivated p-type $In_{0.53}Ga_{0.47}As$ substrates with different numbers of cycles at 150 °C. According to the electrical measurement of the MOS capacitors

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with HfO_2 gate dielectrics (ALD at 200 °C), a minimum capacitance increased with a flat band voltage shift when the number of treatment cycles was increased after an optimal condition. The possible origin for the observed changes in the electrical properties will be discussed based on various characterization results, such as the low temperature C-V measurement and chemical analysis using X-ray photoelectron spectroscopy.

[1] Y.-C. Byun et al., ACS Appl. Mater. Interfaces, 6, 10482 (2014).

[2] A. T. Lucero et al., Electron. Mater. Lett., 11, 769 (2015).

11:00am **TF-TuM10 Electrical Properties of the Atomic-Layer-Deposited** Al₂O₃ on GaSb pretreated with TMA and TDMAT, *Youngseo An, C. Lee, S. Choi, J. Song,* Sungkyunkwan University, Republic of Korea; *Y.-C. Byun, J. Kim,* University of Texas at Dallas, USA; *H.S. Kim,* Sungkyunkwan University, Republic of Korea

GaSb is one of the possible candidates for a p-channel layer in the highspeed metal-oxide-semiconductor field-effect transistors (MOSFETs). However, its poor thermal stability and high reactivity with ambient air produce a poor interface with the high-k gate dielectrics, and there have been several attempts to resolve these problems [1-3].

In this presentation, the sample loading temperature was varied to find an optimal atomic layer deposition (ALD) condition of an Al₂O₋₃ film on p-type GaSb substrates. Then, we compared in situ substrate treatments of two metal precursors, trimethylaluminum (TMA) and tetrakis(dimethylamino)titanium (TDMAT), at the same loading temperature prior to the ALD-Al-2O-3 process. According to various electrical characterizations of the capacitors, lowering the loading temperature was beneficial in reducing the Fermi level pinning effect, and further suppression could be achieved by the subsequent TMA/TDMAT pretreatments. Although the TDMAT treatment showed more efficient relief of Fermi level pinning than the TMA treatment, it undermined the frequency dispersion characteristics in an accumulation region, as an adverse effect. The interface characterization results using X-ray photoelectron spectroscopy will also be discussed to understand the observed electrical properties.

[1] A. Nainani et al., J. Appl. Phys., 109, 114908 (2011).

[2] M. Yokoyama et al., Appl. Phys. Lett., 106, 122902 (2015).

[3] L. B. Ruppalt et al., Appl. Phys. Lett., 101, 231601 (2012)

11:20am TF-TuM11 Using Surface Chemistry for Morphological Control of Metal Oxides and Chalcogenides on Organic Surfaces, J.K. Hedlund, Z. Shi, Amy Walker, University of Texas at Dallas

We describe recent progress in our laboratories to build robust complex two- and three-dimensional composite metal oxide and chalcogenide molecular constructs. This work has important applications in photovoltaics, molecular and organic electronics, sensing, photonics and other technologies. Several recent developments will be discussed including the chemical bath deposition of ZnO transition metal dichalogenides, the atomic layer deposition of ZnO and ZnS and the formation of semiconducting nanowires and other nanostructures on micron-scale patterned surfaces. Optimization and further development of these techniques requires a detailed understanding of the reaction pathways involved in the interaction of organic thin films with organometallic compounds, ions, and other compounds in both solution and gas phases.

11:40am TF-TuM12 Ultra-High-Density Arrays of Defect-Free AlN and GaN Nanorods: A "Space-Filling" Approach for Heteroepitaxial Thin Film Growth, M. Conroy, Pacific Northwest National Laboratory; *Haoning Li*, V. Zubialevich, J. Holmes, P. Parbrook, Tyndall National Institute

Nanostructured semiconductors have a clear potential for improved optoelectronic devices, such as high-efficiency light-emitting diodes (LEDs). However, most arrays of semiconductor nanorods suffer from having relatively low densities (or "fill factors") and a high degree of nonuniformity, especially when produced by self-organized growth. Ideally an array of nanorods for an optoelectronic emitter should have a fill factor close to 100%, with uniform rod diameter and height. In this paper we present a "space-filling" approach for forming defect-free arrays of AIN nanorods, whereby the separation between each rod can be controlled to 5 nm due to a self-limiting process. These arrays of pyramidal-topped AIN nanorods formed over wafer-scale areas by metal organic chemical vapor deposition provide a defect-free semi-polar top surface, for potential optoelectronic device applications with the highest reported fill factor at 98%. We then applied this method to GaN, in particular investigating the

desorption factor that was not an issue for the AIN growth. Here we show that the etched nanorods change morphology during the annealing step even before additional GaN is deposited, forming 6 non-polar side walls.

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