

Friday Morning, November 1, 2013

Thin Film

Room: 104 A - Session TF+EM+NS+SS-FrM

Thin Film: Growth and Characterization III

Moderator: M.R. Davidson, University of Florida

8:40am **TF+EM+NS+SS-FrM2 Atomically Controlled, Self-Limiting Procedures for Growth of Aluminum Oxide on SiC-on-Si to Yield an Atomically Sharp Interface.** *P. Morgen, R. Dhiman, J. Hvam*, University of Southern Denmark, *A. Gomes Silva*, Universidade Nova de Lisboa, Portugal, *K. Pedersen*, Aalborg University, Denmark, *Z. Li*, Aarhus University, Denmark

Electronic devices fabricated from SiC- or SiC/Si epitaxial wafers will need surface passivation and insulating coatings. In earlier work [1], we described a procedure for forming thin (1 nm) Al-oxide layers on Si surfaces, at processing temperatures of around 600°C or slightly higher, resulting in an atomically sharp interface and no silicate formation.

We have adopted the same procedures to a system of SiC/Si, formed by a remote CH₄ plasma interacting with heated Si surfaces in UHV [2]. After growing the SiC/Si system (SiC thickness between 0.5 and 25 nm; polycrystalline) a self-limiting Si-oxide layer was grown on the surface, with a thickness of around 1 nm, at 700°C. On top of this layer we deposited approximately 1 nm of Al with a Knudsen atomic source (all steps in UHV) and then reacted it thermally (at 600°C) with the Si-oxide. We monitored all the process steps and the resulting structures of the layers and the interface using synchrotron radiation induced core level photoemission at ASTRID, Aarhus, Denmark. We found similar qualities with this procedure, as for Si, i.e. an atomically sharp interface between Al-oxide and SiC, which is better than for conventional ALD. This reaction scheme offers self-limiting behavior both of the oxidation to create Si-oxide, and of the conversion into Al-oxide, which only needs a sufficient amount of Al to affect the total conversion of the Si-oxide, while excess Al will leave the system at sufficiently elevated temperatures.

[1] Nanostructured Materials in Different Dimensions for Sensing Applications, *Per Morgen, Joanna Maria Drews, Rajnish Dhiman, and Peter Nielsen*, in: NATO Science for Peace and Security Series B: Physics and Biophysics, Vol. 2011, Nr. 8, 2011, 257-273.

[2] Growth of thin SiC films on Si single crystal wafers with a microwave excited plasma of methane gas, *Rajnish Dhiman, and Per Morgen*, Thin Solid Films, in press 2013, [http://dx.doi.org/10.1016/j.tsf.2013.03.090]

9:00am **TF+EM+NS+SS-FrM3 Understanding Growth Mechanisms of Pyrolytic Graphite by Chemical Vapor Deposition.** *K. Yu, C. Hayman*, Case Western Reserve University, *W. Fan*, Momentive Performance Materials, Inc., *I.T. Martin, H. Martin, R.M. Sankaran*, Case Western Reserve University

Pyrolytic graphite (PG) is a semi-crystalline form of carbon with wide-ranging electronic and thermal applications.^{1,2} The unique anisotropic and unparalleled electronic and thermal properties of PG stem from the alignment of its planar graphite sheets. A variety of structural forms of PG are currently commercially produced by high temperature (>1500 °C) hot-wall chemical vapor deposition (CVD). The degree of alignment (i.e. crystallinity) of the graphite in three dimensions and the growth rate, both of which affect the material properties and cost, depend discretely on process conditions, including growth temperature, gas chemistry, flow geometry, and pressure.

We have recently built a laboratory-scale, high-temperature vacuum reactor to study PG growth. Mixtures of methane and hydrogen gas are reacted to deposit as a carbon film on a heated substrate (1100-2000 °C). Solid carbon deposition under different conditions has been examined to understand the interplay of gas-phase chemistry and substrate nucleation. As-deposited materials are characterized by a suite of analytical techniques. Cross-sectional SEM provides visualization of the interaction of the carbon film with the substrate. The degree of alignment is assessed by micro Raman spectroscopy and X-ray diffraction (XRD). The Raman intensity ratio of the disordered carbon (D) to graphitic carbon (G) bands, an indicator of defects, decreases with increasing deposition temperature, consistent with commercial samples. Perfect Bernal stacking is only found for PG deposited at >2000 °C, as revealed by the 2D band. XRD also reveals the crystallite size through a Scherrer analysis of the peak widths. Smaller crystallite size and larger d-spacing are observed for PG samples deposited at lower temperature. In this talk, we will use discussion of these results to provide a picture of the mechanisms behind graphite nucleation and the PG growth during thermal CVD.

1. *Materials for Aircraft, Missiles and Space Vehicles; Symposium on Materials for Aircraft, Missiles, and Space Vehicles*; the 4th Pacific Area National Meeting: Los Angeles, 1962.

2. Flynn, S. B. *Using Annealed Pyrolytic Graphite in Conduction Cooled Electronics Cooling Applications*; Carleton University: Canada, 2006.

9:20am **TF+EM+NS+SS-FrM4 Characterization of the Thin Films in the Microfabrication of Carbon-Nanotube Templated Thin-Layer Chromatography Plates.** *S. Kanyal, A. Diwan*, Brigham Young University, *D.S. Jensen, A. Dadson, M. Vail*, Diamond Analytics, *M.R. Linford*, Brigham Young University

We have recently described the microfabrication of planar separation devices (thin layer chromatography plates).¹⁻³ These devices are created through the following steps, where silicon wafers are used as the substrates: deposition of 35 nm of alumina, deposition of ca. 6 nm of Fe, annealing of the Fe under a reducing environment to make Fe nanoparticles, carbon nanotube (CNT) growth, conformal coating of the CNTs with SiO₂, oxidative removal of the CNTs, and hydration of the SiO₂ surface. To understand our materials, we have exhaustively characterized them by a suite of characterization techniques. Spectroscopic ellipsometry has taught us about the optical properties of the materials, e.g., thicker Fe films are found to be more metallic than thinner Fe films and alumina films can be well parameterized by a Cauchy dispersion model. ToF-SIMS shows that as surface hydration became more effective, the SiOH⁻/Si⁺ ratio increases. Indeed, the SIMS results are correlated to the more commonly used FTIR analyses, but they appear to have greater predictive power for highly hydrated surfaces. ToF-SIMS also suggests an interesting even/odd effect in ion yields from the CNTs. Rutherford backscattering suggests an interesting potential channeling phenomenon in the CNT forests. XPS shows that the thinner Fe films are completely oxidized and that the CNT surfaces are entirely carbon with only slight oxidation/contamination one year after their fabrication. TEM confirms the deposition of inorganic materials around individual CNTs. AFM shows that the size of the Fe nanoparticles increases with increasing Fe thickness.

1. Jensen, D. S.; Kanyal, S. S.; Gupta, V.; Vail, M. A.; Dadson, A. E.; Engelhard, M.; Vanfleet, R.; Davis, R. C.; Linford, M. R., 195-203.

2. Kanyal, S. S.; Jensen, D. S.; Miles, A. J.; Dadson, A. E.; Vail, M. A.; Olsen, R. E.; Fabien, S.; Nichols, J.; Vanfleet, R.; Davis, R.; Linford, M. R., (3), 031203.

3. Song, J.; Jensen, D. S.; Hutchison, D. N.; Turner, B.; Wood, T.; Dadson, A.; Vail, M. A.; Linford, M. R.; Vanfleet, R. R.; Davis, R. C., (6), 1132-1139.

9:40am **TF+EM+NS+SS-FrM5 A Carbon/Ternary Alloy/Carbon Optical Stack on Mylar as an Optical Data Storage Medium to Potentially Replace Magnetic Tape.** *H. Wang, R. Gates, B. Lunt, M. Asplund*, Brigham Young University, *S. Vaithiyalingam*, Pacific Northwest National Laboratory, *R.C. Davis, M.R. Linford*, Brigham Young University

We have prepared a novel write-once-read-many (WORM) optical stack on Mylar tape as a replacement for magnetic tape. This approach has the potential to substantially increase the longevity of tape data storage, which is extremely important for many large organizations. This work follows our efforts to develop an archival DVD that will last 1000 years (see www.mdisc.com). This optical tape is modified with a ca. 40 nm write layer prepared by co-sputtering bismuth and a tellurium-selenium alloy target to prepare a bismuth-tellurium-selenium (BTS) write layer. This film is sandwiched between thin, protective films of reactively sputtered carbon. The adhesion of this stack to the substrate has been confirmed. Film thicknesses were determined by AFM step height measurements and film composition, morphology, and chemistry were confirmed by RBS, XPS, ToF-SIMS, and SEM. Sub 3 um marks can be made on the Mylar/C/BTS/C tape using 532 nm laser pulses. No marks are observed on the uncoated (control) Mylar substrate under identical conditions. Marks, which showed craters/movement of the write material, were characterized by optical microscopy and atomic force microscopy (AFM). The threshold power for marking the tape was explored as a function of film thickness. Interestingly, the threshold power is higher for either thinner or thicker films and lowest for films of intermediate thicknesses. These results are attributed to a tradeoff between lower optical absorption by thinner films and the requirement of higher powers to melt thicker films. The BTS films appear to be quite stable to storage in the air. The effects of the microscope objective in our home-built apparatus were explored and smaller, higher quality marks could be made with the 60X objective compared to the 40X.

The write process to the film was modeled by COMSOL, and the simulation was consistent with experimental results.

10:00am **TF+EM+NS+SS-FrM6 Toughness Enhancement in Hard Single-crystal $V_{0.5}Mo_{0.5}N/MgO(001)$ Thin Films**, *H. Kindlund, J. Lu*, Linköping University, Sweden, *I.G. Petrov, J.E. Greene*, University of Illinois at Urbana Champaign, *L. Hultman*, Linköping University, Sweden

Hardness is an essential property for a wide range of applications, while for some purposes one also needs ductility to avoid film failure in ceramic films exposed to high stresses. Using VN as a model system, we demonstrate that VMoN alloys exhibit not only enhanced hardness, but impressive increased ductility, i.e. toughness, and study the effect of N vacancies on mechanical properties of pseudobinary NaCl-structure transition metal nitride, VMoN.

$V_{0.5}Mo_{0.5}N_x$ thin films with $0.55 \leq x \leq 1.03$, as determined by RBS, are deposited on MgO(001) substrates by dual reactive magnetron sputtering. For the entire N composition range x , we obtain single-crystal B1-structure $V_{0.5}Mo_{0.5}N_x$, as determined by XRD and TEM analyses. RLM results indicate that the films are relaxed with lattice parameters varying from $a = 4.12 \text{ \AA}$ for $x = 0.55$ to $a = 4.19 \text{ \AA}$ for $x = 1.03$, increasing with increasing N content. The nanoindentation hardness H increases as N-vacancy concentration increases, from $H = 18 \text{ GPa}$ ($x=1.03$), to 20 GPa ($x=0.94$), 21 GPa ($x=0.72$), to 26 GPa ($x=0.55$), while the elastic modulus is maintained essentially constant. In addition, while nanoindented VN and TiN reference samples suffer from severe cracking typical of brittle ceramics, $V_{0.5}Mo_{0.5}N_x$ films do not crack. Instead, they exhibit material pile-up around nanoindenters, characteristic of plastic flow in ductile materials.

10:20am **TF+EM+NS+SS-FrM7 Manganese-based Thin Films Growth on Silicon Oxide Substrates**, *H. Sun, X. Qin, F. Zaera*, University of California, Riverside

The growth of manganese-based films on silicon oxide substrates via the chemical vapor decomposition of two Mn metalorganic precursors, methylcyclopentadienylmanganese(I) tricarbonyl, $MeCpMn(CO)_3$, and bis(N,N' diisopropylpentylamidinato)Mn(II), was characterized and contrasted by X-ray photoelectron spectroscopy (XPS). $MeCpMn(CO)_3$ proved to be much less reactive than the acetamidinate, even if gas-phase activation was used to promote the growth of Mn films at lower temperatures in the first case. The acetamidinate precursor does show high reactivity, affording the deposition of Mn at reasonable rates, higher at higher temperatures, but also leads to the incorporation of approximately 15% of nitrogen and additional carbon in the grown Mn(0) films. In both depositions, a nonstoichiometric mixture of $MnO_x + SiO_x$ and Mn silicate is formed first, possibly followed by the formation of a thin subsurface Mn silicide layer. Mn(0) metallic films can be grown on top of the combined Mn silicate/Mn silicide structure, which serves as an effective diffusion barrier. The Mn(0) species deposited on top of those initial layers could be oxidized by N_2O or O_2 , during which the organic ligands could be effectively removed from the surface. As a result, manganese-based thin films growth could be achieved by repeating this deposition-oxidation process.

10:40am **TF+EM+NS+SS-FrM8 Properties of Epitaxial $VN_x/MgO(001)$ ($0.70 < x < 1.36$) Layers Grown by Reactive Magnetron Sputter Deposition**, *A.R.B. Mei*, University of Illinois at Urbana Champaign, *D.G. Sangiovanni, H. Kindlund*, Linköping University, Sweden, *B. Howe*, Air Force Research Laboratory, *E. Broitman, V. Chirita, L. Hultman*, Linköping University, Sweden, *A. Rockett, J.E. Greene, I.G. Petrov*, University of Illinois at Urbana Champaign

Ceramic coatings are, despite their high hardness, often limited in use by their brittleness. Under high stress conditions, brittle hard coatings, such as transition metal (TM) nitride thin-films, fail prematurely through crack nucleation and growth. In order to extend the longevity and broaden the applications of ceramic hard coatings, they must be toughened: their capacity for absorbing elastic energy prior to fracture must be increased. Here, we report on the toughening of under-stoichiometric VN_x , deposited on MgO(001) in ultra-high-vacuum by reactive magnetically-unbalanced magnetron-sputter deposition. X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning transmission electron microscopy (STEM), and selected area electron diffraction (SAED) indicate that the films are fully-dense single-phase epitaxial $VN_x(001)$. Hardness values of $VN_x(001)$, determined from nanoindentation measurements according to the Oliver and Pharr method, increase with off-stoichiometry from $H = 14.0 \pm 0.8 \text{ GPa}$ ($x = 1$) to $17 \pm 0.76 \text{ GPa}$ ($x = 0.77$) and $16.7 \pm 0.1 \text{ GPa}$ ($x = 1.20$). Scanning electron micrographs (SEM) of nanoindentations performed with a sharp cube-corner tip reveal the presence of cracks in $VN_x(001)$ overlayers with $x > 0.95$; four cracks were observed in each film with $x > 1.23$, two in films between $1.06 < x < 1.10$, and one for $0.95 < x < 1.00$. Cracks were not observed in $VN_x(001)$ with $x < 0.88$. The resilience of

highly under-stoichiometric $VN_x(001)$ films to fracturing, as indicated by the absence of cracks, is evaluated via crystal orbital overlap populations (COOP) analysis using *ab initio* density functional theory (DFT) calculations. The results indicate that the toughening is of electronic origin and due to the strengthening of V-V bonds along the slip direction.

11:00am **TF+EM+NS+SS-FrM9 Sandwiched Antibacterial Layers Topped by Thin Polymeric Coatings and Deposited in Thin Capillaries**, *G.F. Franz, F.S. Schamberger*, Munich University of Applied Sciences, Germany

Bottom-down strategies for nanostructures require tailoring techniques for very thin layers. One of the film-building polymers which cannot be deposited with ALD is poly-p-xylylene, commonly known as polyparylene, which has evolved to generate universally employable layers with unique properties. As known from growth theory, very thin layers below 100 nm in thickness are necessarily porous which reduces the property of preventing diffusion. This feature, however, is required to control the process of dissolution from metallic silver to silver ions of a layer lying beneath. The silver layer is used as a long-term depot for the generation of silver ions which exhibit an antibacterial conduct. The silver ions break through the cell walls of the bacteria, causing them to die.

To deposit very thin layers of polyparylene, the conventional Gorham method has been improved. We present a completely new method to control the growth of these very thin layers with defined porosity for which exact knowledge of vapor pressure and evaporation rate is required [1]. To tailor this long-term property above the death-causing threshold, the porosity (i. e. hole density) is:

(i) quantitatively determined with a digital evaluation procedure of the micrographs gained with AFM as function of the parameters of deposition.

and (ii) has been correlated with the breakdown voltage and the capacitance which can both easily applied to the samples (electro impedance spectroscopy, EIS).

This makes the hole density a quantitative measure to rate the degree of permeability for silver ions. Applying ICP-OES, the concentration of silver ions in the adjacent liquid around the silver spots is measured. This concentration depends on the thickness of the films of polyparylene. With standard biological procedures, this is correlated to the lethal impact on bacteria. Whereas layer growth upon open surfaces is controlled by the density of reactive particles (surface polymerization), the density becomes locally dependent for coating of narrow holes which occurs as a diffusive process with losses due to deposition. In a series of experiments, the growth behavior in thin, narrow pipes with an aspect ratio between 10 and 30 has been obtained, and a theoretical model is presented which reflects the crossover of these two transport mechanisms as function of chamber pressure and temperature.

[1] G. Franz et al., German patent disclosure DE 2012 014 915.8

11:20am **TF+EM+NS+SS-FrM10 Wurtzite Phase Stable $Mg_xZn_{1-x}O$ Epilayers with High Mg Compositions for High Performance Ultraviolet Photodetectors**, *M. Wei, R.C. Boutwell*, University of Central Florida, *N. Faleev*, Arizona State University, *A. Osinsky, R. Miller*, Agnitron Technology Inc., *W.V. Schoenfeld*, University of Central Florida

Wide band gap semiconductors such as $Mg_xZn_{1-x}O$ represent an excellent choice for making optical photodetectors and emitters operating in the UV spectral region. High crystal and optical quality $Mg_xZn_{1-x}O$ thin films were grown epitaxially on c-plane sapphire substrates by plasma-assisted Molecular Beam Epitaxy. ZnO thin films with high crystalline quality, low defect and dislocation densities, and sub-nanometer surface roughness were achieved by applying a low temperature nucleation layer. The critical growth conditions were discussed to obtain a high quality film: the sequence of Zn and O sources for initial growth of nucleation layer, growth temperatures for both ZnO nucleation and growth layers, and Zn/O ratio. Resultant epitaxial ZnO films demonstrated a root-mean-square surface roughness of 0.373 nm for $1 \mu\text{m} \times 1 \mu\text{m}$ atomic force microscope images with clear hexagonal shaped terrace steps. The x-ray diffraction FWHM for (0002) peak was measured to be 13 arc sec for ZnO. By tuning Mg/Zn flux ratio, wurtzite $Mg_xZn_{1-x}O$ thin films with Mg composition as high as $x=0.46$ were obtained without phase segregation. The steep optical absorption edges were shown with a cut-off wavelength as short as 278 nm , indicating of suitability of such material for solar blind photo detectors. Consequently, Metal-Semiconductor-Metal photoconductive and Schottky barrier devices with interdigital electrode geometry and active surface area of 1 mm^2 were fabricated and characterized. Photoconductor based on $Mg_{0.46}Zn_{0.54}O$ showed $\sim 10^2 \text{ A/W}$ peak responsivity at wavelength of $\sim 260 \text{ nm}$. The spectral cutoff of the devices was close to 315 nm with more than two orders of magnitude visible rejection ratio ($R_{260 \text{ nm}}/R_{400 \text{ nm}}$) these devices good candidates for solar blind applications.

11:40am **TF+EM+NS+SS-FrM11 Self-assembling Monolayers on Oxides: Utopia?**, T. Hauffman, A. Hubin, H.A. Terryn, Vrije Universiteit Brussel, Belgium

Self-assembling monolayers are one of the most innovative ways to functionalise and change material surface' properties on the nanoscale. The adsorption of thiols on gold has proven the feasibility to build such well-ordered, compact monomolecular organic structures. However, gold is industrially less relevant and the variety of organic moieties that can be deposited in this way on the noble substrate is very limited.

Therefore, an interesting approach is outlined in literature, involving the deposition of monolayers on oxide films. In particular, the adsorption of phosphonic acids on aluminium oxides is proposed. In this presentation, we address the adsorption of n-octylphosphonic acids from aqueous and ethanolic solutions. It could be proven that not only the adsorbed layer should be characterised, but that the bare sample and the solvent play a crucial role as well. Furthermore, interactions between the oxide, the solvent and the individual molecules should be considered.

In this work, a strong focus laid on the in situ, real time monitoring of the dynamic adsorption of the organic molecules. Therefore, a methodology including in situ AFM, in situ, visual ellipsometry and Odd Random Phase Electrochemical Multisine Impedance Spectroscopy is outlined. Ex situ complementary techniques, such as XPS and FE SEM were included.

As such, it could be proven that in polar solvents, molecules adsorb in a fast and Stranski-Krastanov way. Moving to less polar solvents, such as ethanol, provoke deterioration of the oxide surface. The phosphonic acids adsorb on the surface, giving rise to condensed water on the surface. The acid molecules dissolve in this water, creating a hazardous environment for the oxide film.

In general, we state that it is impossible to form a SAM on aluminium oxide through an acid base elimination condensation reaction using organic molecules that are soluble in water [1-5]. Moreover, conformation and density issues are to be expected when using longer, non water soluble phosphonic acid.

References

1. T. Hauffman, Y. Van Ingelgem, T. Breugelmans, E. Tourwé, H. Terryn, A. Hubin, *Electrochimica Acta*, accepted for publication.
2. T. Hauffman, A. Hubin, H. Terryn, *Surface and Interface Analysis* DOI 10.1002/sia.5150.
3. T. Hauffman, T. Breugelmans, Y. Van Ingelgem, E. Tourwé, H. Terryn, A. Hubin, *Electrochemistry Communications* 22 (2012) 124-127.
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5. T. Hauffman, O. Blajiev, J. Snauwaert, C. Van Haesendonck, A. Hubin, H. Terryn, *Langmuir* 24 (2008) 13450-13456.

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