

Wednesday Morning, October 31, 2012

Thin Film

Room: 10 - Session TF-WeM

Thin Films for Encapsulation, Packaging, and Biomedical Devices

Moderator: L.W. Rieth, University of Utah

8:00am **TF-WeM1 Optimizing a Spatial Atomic Layer Deposition Cell for High Throughput, Low Temperature, Roll-to-Roll Applications.** *M.J. Dalberth, L. Lecordier, M.J. Sershen, M. Ruffo, R. Coutu, G. Sundaram, J.S. Becker*, Cambridge Nanotech, Inc.

Atomic layer deposition (ALD) has established itself as a technique capable of producing uniform, dense, pin-hole free films with extremely fine thickness control. The surface reactions involved in many ALD processes are thermally active at temperatures less than 200°C which makes it attractive for emerging applications coating substrates with a limited thermal budget- for example, a barrier layer for organic photovoltaics on a flexible substrate. Many of these applications demand high throughput, however, and traditional ALD is too slow due to its temporally spaced pulses of reactant A and reactant B, and its need for a vacuum system requiring time consuming evacuation. Spatial ALD replaces the temporal separation of reactants with their spatial separation by confining them to separate gas channels in a deposition cell. Plus, it's a process that takes place at atmospheric pressure and can eliminate system evacuation times. In spatial ALD, the substrate and cell move relative to one another, and the number of A/B channels determines the thickness of material deposited. At speeds of 10m/min or more, equivalent throughput of 2000-4000 wafers can potentially be achieved with equivalent cycle time $\ll 0.1s/\text{\AA}$. As of today, Cambridge Nanotech has implemented two 150 mm x 150 mm cells based on two and six-cycle designs. 150mm Si wafer and PEN/PET substrates were processed using TMA and water at atmospheric pressure, 100-120°C, 10m/min speed and 0.1-0.5 mm gap size between cell and substrate. The impact of key process metrics on process performance such as GPC or uniformity was evaluated, including reactant dosage, temperature or stage velocity. Not surprisingly, the ability to maintain the spatial confinement of TMA and water in order to limit parasitic CVD-like reactions (which induces higher GPC and particle formation) is shown to be critically dependent on gap size and flow rate for the inert gas barriers. Data showing the impact of different cell designs (e.g., orifice distribution and size) on wafer- and process-scale metrics will also be discussed. While development on new cells is continuing, the data taken so far support the outlook that spatial ALD could be a key technology for rapid deposition of functional layers in high throughput applications.

8:20am **TF-WeM2 Encapsulation of Implantable Devices by Atomic Layer Deposited Al_2O_3 and Parylene C Bi-layer.** *X. Xie, L.W. Rieth, F. Solzbacher*, University of Utah

Encapsulation of 3-D electronic biomedical implants with complex geometries and tight gaps between components is one of the greatest challenges to achieve long-term functionality and stability. We have investigated a new encapsulation scheme that combines atomic layer deposited (ALD) Al_2O_3 and Parylene C for biomedical implantable system and will present results to quantify the performance of this system. Our approach combines the highly effective moisture barrier properties of ALD alumina, and Parylene as a barrier to many ions and for preventing contact of alumina with liquid water. 52 nm of Al_2O_3 was deposited by plasma-assisted (PA) ALD on interdigitated electrodes (IDEs). AFM micrographs show that as-deposited Al_2O_3 films on fused silica substrate have RMS surface roughness of 0.48 nm. XPS spectra determined that PA-ALD films had nearly stoichiometric O/Al ratio of 1.4. A 6- μm thick Parylene-C layer was deposited by CVD using Gorman process on top of Al_2O_3 and used A-174 (Momentive Performance Materials), an organosilane, as adhesion promoter. The IDEs coated with Al_2O_3 -Parylene C were soaked in phosphate buffered saline (PBS) solution for a period of about 9 months at both body temperature (37°C) and elevated temperatures (57 to 80°C) for accelerated lifetime testing. Electrochemical impedance spectroscopy (EIS) and chronoamperometry were used to evaluate the integrity and insulation performance of the soft encapsulation. The leakage current was ~ 20 pA by applying 5 V DC bias and impedance was ~ 3.5 M Ω at 1 kHz with phase of close to -87° by using EIS for samples under 67°C about 9 months (approximately equivalent to 72 months at 37°C), indicating no significant degradation. The encapsulation performances of combining alumina and Parylene C, Parylene C only and alumina only coatings were compared and the bi-layer coating shows its superiority of at least 5 times longer lifetime than the rest two coating approaches. The continuous 5 V bias voltage has

no obvious effect on alumina and Parylene coated samples while it shortened the lifetime of Parylene coating by at least a factor of 4. Complex topography can shorten the lifetime of coating dramatically comparing with planar structures, especially with the existence of micromotion inside the body. The lifetime of alumina and Parylene coated devices with hand-wound coils and SMD capacitors was only about 50% or less of that of planar test structures. The long-term (more than 6 years of equivalent lifetime) insulation performance of the novel double-layer encapsulation shows its potential usefulness for chronic implantable electronic microsystems.

8:40am **TF-WeM3 Ultra-barrier Technology for Moisture-Sensitive Electronics.** *P.F. Carcia*, DuPont Central Research and Development

INVITED

Organic materials are driving future electronic technologies in the direction of lighter weight, more robust, flexible, and lower cost manufacturing. Organic solar cells have reached 10% efficiency in the lab and are becoming an attractive low-cost option for generating electricity in isolated regions in the undeveloped world. Organic light emitting diodes (OLEDs) are poised to succeed LCDs as the next generation of more vibrant color displays for TVs, computer tablets, and mobile phones. However, one obstacle to the broad utilization of organic electronic materials, and also some inorganic materials, is their sensitivity to atmospheric gases, especially moisture. In the case of OLED displays, hermetic packaging with glass or metal may be an interim solution for early product introduction, but this precludes advantages of lighter weight and flexibility, which could have cost penalties. But for organic photovoltaics (OPV), and for some thin-film inorganic PV technologies, e.g. CIGS, to be cost competitive, they will need to be fabricated with flexible polymer materials, which are permeable. These polymer materials will require low permeation coatings to exclude atmospheric gases that can readily degrade moisture sensitive electronic materials. For OLEDs, the barrier coating must uniformly reduce permeation through the polymer by $\sim 10^6$ times for the display to be defect-free. Solar cells will need a similar (10^4 - 10^5 times) reduction in permeation, if they are to have a reasonable outdoor lifetime. In our laboratory, we have discovered that single-layer, inorganic, barrier coatings grown by atomic layer deposition (ALD) can meet the demanding requirements of an ultra-barrier to protect sensitive electronic devices on a flexible polymer substrate. In this talk we will discuss many of the materials and processing insights we have learned about ALD barriers, their performance protecting sensitive electronic devices, and the outlook for practical, high-speed ALD manufacturing.

9:20am **TF-WeM5 Multilayer Barrier Coatings for Organic Photovoltaics.** *A.M. Coclite, F. De Luca, K.K. Gleason*, Massachusetts Institute of Technology

Encapsulant barrier coatings, which prevent the permeation of water through flexible plastic substrates, are an enabling technology for the commercialization of OPV devices. Such protective coatings are made of multilayer stacks where multiple dense, inorganic layers are alternated with soft, organic ones. The inorganic layer contains inevitably some pinholes and defects. The roles of the organic layer are creating a tortuous and longer path among the defects of two successive inorganic layers, filling the pores of the inorganic underlayer limiting the propagation of defects from one inorganic layer to the other and smoothing the substrate surface roughness.

We obtained good barrier properties ($\text{WVTR} = 10^{-2}$ g/cm²/day at 25°C, RH=85%) with a bilayer obtained by coupling initiated-PECVD (iPECVD) and plasma enhanced CVD (PECVD) at very low thickness of inorganic layer (25 nm).

SiO_x layers were deposited through PECVD in MW plasma at high power and high oxygen dilution. The silanol and organic groups were not detectable by IR spectroscopy, resulting in dense film with high flexibility and high critical tensile strain. High critical tensile strain implies that the coating can be bent and stretched to a relatively big extent before cracking. Inorganic films obtained by other technologies (i.e. Al_2O_3 ALD coatings) showed smaller critical strain values.

Organic coatings were deposited through a new process named iPECVD with enhanced monomer structure retention compared to a conventional plasma deposition and faster deposition rate if compared to conventional iCVD processes from organosilicon monomer. The deposition conditions were tuned to obtained good planarizing properties. The deposition of planarizing organic layers was demonstrated by depositing the coating on the top of some microspheres (1 μm in diameter) which served as model defects on the surface. Increasing the thickness of the coating, the degree of planarization (DP), both local (DLP) and global (DGP), increases. The DLP

increases much faster than the DGP: when the coating is 1 μm -thick the DLP is already 99%, for the global planarization instead a 1.8 μm -thick-coating is needed to reach DGP= 99%.

The great advantage of a similar approach is that we deposit the multilayer in a large-area reactor, maintaining the same organosilicon precursor and the same reactor configuration for both deposition of silica-like and organosilicon layers. A detailed investigation of the barrier and mechanical properties changing the number of layers in the stack and the measurements conditions will be presented in order to demonstrate the robustness of the following approach to create flexible ultra-high barrier layer.

9:40am **TF-WeM6 Preparation of Hydrophobic Coatings on Si/SiO₂ by Incorporation of Nano- and Microdiamond in a Layer-By-Layer Deposition.** *A. Diwan, J. Wilcock, M.R. Linford*, Brigham Young University

Hydrophobic coatings are required in a wide variety of applications. We are preparing robust hydrophobic coatings on silicon from nano-/microdiamond and polyallylamine (PAAm) deposited in a layer-by-layer fashion. After deposition of PAAm/diamond multilayers, two different approaches were considered for preparing hydrophobic surfaces. The first involved the reaction of the amine groups of PAAm with the epoxy groups of 2-(1H,1H-perfluoroundecyl)oxirane to yield hydrophobic surfaces. However, due to the formation of hydroxyl groups in this ring opening reaction, the roll off of water drops, i.e., their receding contact angles, was poor although the surface showed high static and advancing water contact angles. The other approach, which appears to produce more robust coatings, used only nano-diamond and PAAm for the growth of electrostatically bound layers. The amine of the PAAm was reacted with (3-glycidyloxypropyl)trimethoxysilane (GPTMS) in a closed pressure vessel at 100°C, which should provide crosslinking to the film and also active sites for subsequent reaction. The final step involved the chemical vapor deposition of a fluorinated silane (F-13 silane) to give a hydrophobic surface. The static water contact angle of a flat PAAm/GPTMS/F-13 silane surface was 107°, which showed good roll off properties. The prepared coatings are evaluated and analyzed at every step using atomic force microscopy (AFM), x-ray photoelectron spectroscopy (XPS), time-of-flight secondary ion mass spectroscopy, ellipsometry for thickness measurements and water contact angles measurements.

Authors Index

Bold page numbers indicate the presenter

— B —

Becker, J.S.: TF-WeM1, 1

— C —

Carcia, P.F.: TF-WeM3, **1**

Coclite, A.M.: TF-WeM5, **1**

Coutu, R.: TF-WeM1, 1

— D —

Dalberth, M.J.: TF-WeM1, **1**

De Luca, F.: TF-WeM5, 1

Diwan, A.: TF-WeM6, 2

— G —

Gleason, K.K.: TF-WeM5, 1

— L —

Lecordier, L.: TF-WeM1, 1

Linford, M.R.: TF-WeM6, 2

— R —

Rieth, L.W.: TF-WeM2, **1**

Ruffo, M.: TF-WeM1, 1

— S —

Sershen, M.J.: TF-WeM1, 1

Solzbacher, F.: TF-WeM2, 1

Sundaram, G.: TF-WeM1, 1

— W —

Wilcock, J.: TF-WeM6, 2

— X —

Xie, X.: TF-WeM2, 1