

Thursday Afternoon Poster Sessions

Thin Film

Room: Central Hall - Session TF-ThP

Thin Film Poster Session

TF-ThP1 Vanadium Oxide Thin Films Grown by ALD using TEMAV and O₃ or H₂O Precursors. A. Premkumar, IMEC, Belgium, M. Toeller, Tokyo Electron Limited, Japan, I. Radu, Katholieke Universiteit, Leuven, Belgium, C. Adelman, M. Schaeckers, J. Meersschaut, T. Conard, J. Malgorzata, S. Van Elshocht, IMEC, Belgium

Vanadium dioxide (VO₂) is a smart material and offers interesting optical and electrical switching applications because of the reversible semiconductor-metal transition (SMT) that is observed at a temperature of about 68°C. Thin film growth of VO₂ has been extensively studied by CVD, PVD and sol-gel techniques. Aggressive scaling and increasing integration complexity have placed greater importance on atomic layer deposition (ALD) for depositing oxides in microelectronics. In this work we have developed an ALD process for VO₂ on 300 mm Si substrates using TEMAV precursor comparing different reactants (O₃ and H₂O) and starting surfaces (SiO₂ or Al₂O₃). We studied the ALD process in a 100-210°C temperature window. The as deposited films (100-150°C) were found to be XRD amorphous. Annealing in N₂/O₂ ambient (425-500°C) resulted in crystalline films. For the O₃ based process, the VO₂ formation conditions were found to be strongly dependent on the substrate investigated with a narrow process window for the preparation of phase pure and continuous VO₂ films. In contrast, highly uniform layers with consistent phase formation pathways were observed for the water based ALD process on all the substrates investigated. Films resulting from the water based process were smoother. Growth/anneal conditions were optimized to yield a resistivity change at the SMT of two orders of magnitude for 8 nm VO_x films.

TF-ThP2 Roles of MoO₃ Layer for Charge Injection and Charge Generation in an Organic Light Emitting Diode. M. Kawamura, S. Yoshida, Y. Abe, Kitami Institute of Technology, Japan

It has been reported that MoO₃ inserted between ITO anode and hole transport layer is useful as a hole injection layer to improve properties of organic light emitting diodes (OLEDs). However, its optimum thickness in the devices varies from sub-nanometer to 30 nm, depending on reports. In addition, as the mechanism of hole injection at the MoO₃ layer and α -NPD layer, it is argued that either charge injection mechanism or charge generation mechanism is dominant. As a fundamental study to investigate the main mechanism, we investigate the influence of MoO₃ thickness on properties of normal OLED device and also a device with a charge generation layer in the present work. First of all, we investigated properties of an OLED consists of ITO/MoO₃/ α -NPD/Alq₃/LiF/Al prepared by vacuum evaporation. As a results, the best device properties were obtained when 1.0 nm thick MoO₃ was inserted. Using atomic force microscopy, we found that the best device properties were obtained when the ITO surface was not completely covered with MoO₃. It is considered that the incomplete coverage was preferable in the view point of energy alignment because HOMO of α -NPD (5.4 eV) is intermediate between work functions of ITO (5.0 eV) and MoO₃ (5.7 eV). Then we confirmed charge generation phenomenon using device consists of ITO/Alq₃/MoO₃/ α -NPD/Al. Current did not flow without MoO₃ layer. When thickness of the MoO₃ layer was above 1.0 nm, a large current flowed and the current-voltage curves were the same even the MoO₃ thickness was increased to 10 nm. As the reason, it is considered that a high charge generation ratio was obtained from continuous MoO₃ layer. Consequently, we propose that MoO₃ work mainly for charge injection when the thickness is thin discontinuous film, and for charge generation when thick continuous film.

TF-ThP4 Effects of Preparation Conditions on the Magnetocaloric Effect of Gd Thin Films. H.F. Kirby, D.D. Belyea, J.T. Willman, University of South Florida, C.G. Hendryx, Newsome High School, C.W. Miller, University of South Florida

The effects of deposition temperature, post-deposition annealing, and chamber gettering were investigated on the magnetocaloric properties in Ta(5nm)/Gd(30nm)/Ta(5nm) grown by magnetron sputtering. The magnetocaloric effect (MCE) in these thin films, as indicated by the magnetic entropy change around the ordering temperature, increases with both growth temperature and post-deposition annealing of samples grown under ambient conditions. The full width at half max of the entropy change peak generally decreased toward the value for bulk Gd with increasing

deposition and annealing temperature. Similarly, the temperature of the maximum entropy change increased toward the bulk Gd ordering temperature. Overall the relative cooling power increased as deposition and annealing temperatures were increased. Gettering proved useful in limiting oxidation of the Gd especially for high temperature growth: ungettered samples grown at 600°C were purely diamagnetic GdO (111), while gettered samples, though still containing some GdO, were ferromagnetic.

TF-ThP5 Advanced Analytical Characterization of Multilayered Thin Films for Corrosion Inhibition. G. Zorn, M. Karadge, GE Global Research, C.C. Pierce, J.I. Melzer, GE Power & Water, M.M. Morra, GE Global Research

Advanced corrosion inhibitors developed by General Electric Power and Water can have complex multilayered structures that incorporate metal, ceramic and polymeric structures. For optimal performance it is important to understand the structure, morphology and composition of different layers. However, characterizing these nano scale films is very challenging, as they can be sensitive to preparation technique and damage. Moreover, surface roughness and homogeneity of the layers should be considered. The challenges in characterizing these multilayered structures will be discussed as an example for thin film characterization in the industrial R&D world. A multi technique approach that provides a detailed view of complex structures and compositions will be presented. Transmission Electron Microscopy (TEM) equipped with EDS was used to define local morphologies, crystalline structures and chemical composition; and Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) depth profiles were used to determine the molecular distribution within the different layers. TEM provides local information in the range of a few nanometers over Focused Ion Beam (FIB) cross sections while ToF SIMS allows the analysis of larger areas, in the range of hundreds of nanometers, and provides top down views of the layers. The talk will emphasize how these two methods complement each other to achieve a detailed picture of complex structures within thin films.

TF-ThP6 Sputter Deposition of Atomically Smooth ZnO Films with Buffer Layers Crystallized via Nitrogen Mediation. K. Kuwahara, Kyushu University, Japan

Zinc oxide (ZnO) is a promising oxide semiconductor for optoelectronic devices because of its attractive properties such as wide direct band gap (3.3 eV), high exciton binding energy (58 meV), and material abundance. For realizing optoelectronic devices utilizing such attractive properties, a fabrication method of high quality crystalline ZnO films is essential. We have recently demonstrated a novel fabrication method of ZnO films utilizing nitrogen mediated crystallization (NMC), where the crystal nuclei density can be controlled because the nitrogen atoms suppress crystallization of ZnO films [1]. By using NMC-ZnO films as buffer layers, we have succeeded in high-quality epitaxial growth of ZnO films on sapphire substrates by RF magnetron sputtering [2]. However, for device applications such as light emitting diodes (LED), there still remains a need for improvement of properties of ZnO films. Here we apply off-axis sputtering to epitaxial growth of ZnO films on NMC-ZnO buffer layers aiming at reduction of negative-ion bombardment that causes serious damage during oxide deposition. NMC-ZnO buffer layers were fabricated by RF magnetron sputtering. The used gas was N₂-Ar and the total pressure was 0.3 Pa. ZnO ceramic targets were used. The applied RF power was 100 W and the deposition temperature was 700°C. The thickness of the buffer layers was 10 nm. On NMC-ZnO buffer layers, ZnO films were deposited by off-axis RF magnetron sputtering at 700°C. Ar-O₂ was used and the total pressure was 0.67 Pa. The applied RF power was 60 W. The film thickness was 1 μ m. After deposition, the films were annealed in a furnace at 1000°C for 3h in air. The surface of as-deposited ZnO films fabricated by off-axis sputtering on NMC-ZnO buffer layers has subnm scale corrugation and the RMS roughness is 0.28 nm, being significantly small compared with 1.02 nm for the films fabricated by conventional on-axis sputtering. Moreover, the annealed surface has 0.26-nm-high steps corresponding to one molecular layer of ZnO. We have demonstrated fabrication of atomically flat ZnO films by using NMC-ZnO buffer layers together with the off-axis sputtering.

[1] N. Itagaki, K. Kuwahara, K. Nakahara, D. Yamashita, G. Uchida, K. Koga, and M. Shiratani, Appl. Phys. Express 4 (2011) 011101.

[2] K. Kuwahara, N. Itagaki, K. Nakahara, D. Yamashita, G. Uchida, K. Kamataki, K. Koga, and M. Shiratani, Thin Solid Films 520 (2012) 4674.

TF-ThP7 Influence of Substrate Temperature on the Microstructure and Surface Morphology of Pulsed DC Magnetron Sputtered ZrB₂ Films. *C.T. Lee, W.C. Chen*, Instrument Technology Research Center, Taiwan, Republic of China

The ZrB₂ films were prepared on Si(100) substrate by pulsed DC magnetron sputtering with ZrB₂ target. Effects of substrate temperature (from 400 °C to 550 °C) on the microstructure and surface roughness of ZrB₂ films were investigated by X-ray diffraction, field emission scanning electron microscopy and atomic force microscopy. X-ray diffraction analysis reveals that ZrB₂ film was polycrystalline with (001) and (101) orientation when substrate temperature was 450 °C. However, the ZrB₂ film has preferred orientation along (001) when substrate temperature was above 500 °C. An increase in average grain size with increase of substrate temperature was observed. The average grain size of ZrB₂ film was increased from 6.2 nm to 15 nm as substrate temperature increased from 400 °C to 550 °C. In this study, the preferred orientation along (001) of ZrB₂ films on Si(100) substrate can be obtained at substrate temperature above 500 °C by pulsed DC magnetron sputtering.

TF-ThP8 Effect of Fluorine Doping on the Structural, Optical and Electrical Properties of CdS Films Deposited by Chemical Bath Deposition. *K.E. Nieto-Zepeda, Cinvestav-IPN, Mexico, E. Mota-Pineda, ESIME-IPN, Mexico, M.A. Zapata-Torres, CICATA-Legaria, IPN, Mexico, M.A. Melendez-Lira, Cinvestav-IPN, Mexico*

The efficiency of photovoltaic structures based on the CdS/CdTe heterojunction is far from that predicted theoretically. Within the various problems that affect negatively the efficiency of this system is the lack of a methodology to get a controlled doping of the n-type window layer of CdS. The chemical bath deposition methodology generally produces uncontrolled n-type CdS films. This paper proposes the use of fluoride as an electron donor to substituting the atoms of S in CdS. With this objective we deposited CdS films using different molar compositions of thiourea doped with fluorine. The molar compositions used are between 0.025 and 0.25 M with variations of 0.025 M. The samples obtained were characterized by UV-Vis transmission spectroscopy, X-ray diffraction and the electric transport by I vs V measurements. Representative samples were characterized by using Atomic Force Microscopy, Scanning Electron Microscopy, Raman, photoluminescence and photoconductivity spectroscopies. The results are discussed considering the presence of defects associated with the molar concentration of thiourea. The crystallographic quality of the samples increases with fluorine doping. There is a growing trend in the band gap values with thiourea concentrations. Raman spectroscopy results suggest that fluorine is substitutionally incorporated in sulphur sites and photoluminescence spectroscopy indicates that chemical bath deposited samples have a low density of radiative defects. I vs V curves indicated that transport is carried out through a percolation process. Our results indicated that the samples with the best characteristics are those fluorine doped grown with thiourea concentrations between 0.075 y 0.125 M showing that fluorine substitutionally replaces to sulphur and passivates interfacial states.

* This work has been partially supported by CONACYT-Mexico and Instituto de Ciencia y Tecnologia-DF

TF-ThP9 Interfacial Properties of Atomic Layer Deposited TiO₂ Films on InAs (100) Surfaces. *L. Ye, T. Gougousi*, UMBC

TiO₂ has been deposited on both native oxide and etched InAs (100) surfaces by thermal atomic layer deposition (ALD) from tetrakis dimethyl amido titanium (TDMAT) and H₂O. X-ray photoelectron spectroscopy (XPS) and high resolution transmission electron microscopy (HRTEM) were utilized to study the interface between the TiO₂ films and the InAs substrate. For depositions at 200°C, the native oxide was thinned and part of the native oxide bubbled to the top of the TiO₂ surface. HRTEM data for a ~4 nm film of TiO₂ on InAs confirmed that the native oxide was completely removed from the interface. When TiO₂ was deposited on HF and NH₄OH etched InAs surfaces, practically sharp interfaces were maintained. To investigate the effect of temperature on the native oxide consumption, two sets of samples with film thickness of 2 and 3 nm were prepared at deposition temperatures ranging from 100 to 325°C. XPS showed that the native oxides were consumed most effectively at 250°C. Deviation of the deposition temperature from 250°C in either direction resulted in a reduction of the native oxide consumption rates.

TF-ThP10 AES and XPS Characterizations in ALD ZnO Films Doped with Al and P. *H. Yuan*, Northwestern Polytechnical University, China, *B. Luo, W.L. Gladfelter, S.A. Campbell*, University of Minnesota

Zinc oxide based films are transparent conductive oxide materials. As part of our research in solar cells, Al- and P-doped ZnO films were prepared at 250 °C in a hot-wall atomic layer deposition (ALD) system. Ozone, diethylzinc, trimethylaluminum or trimethylphosphite were used as the

precursors. We obtained films with different Al or P concentrations by varying the precursor vapor pressures, and characterized their physical, chemical and electrical properties. The Al-doped films were n-type with the lowest resistivity occurring at an Al concentration of 1-2%. The as-deposited P-doped films were n-type, but upon rapid thermal annealing in oxygen, the films changed to p-type. The temperature of the n- to p-type transition decreased as the phosphorus concentration increased.

In this presentation, we will describe details of the Auger electron spectroscopic (AES) and X-ray photoelectron spectroscopic (XPS) measurements. AES depth profiling was used to determine the compositions of all of the films, which confirmed that a layered microstructure for the films prepared by introducing the Zn precursor and Al precursor alternatively, and a homogeneous distribution of the Al in the films prepared by co-injecting the Al and Zn precursors. XPS determined the compositional change of the oxygen species upon sputtering. The oxidation state of the P dopant was determined to be +5 for both the as-deposited and annealed films.

TF-ThP12 Photoluminescence and Life-Time Characterization of Polythiophene Incorporated with Dye Molecules. *H. Kobe, H. Kato, A. Yamada, S. Takemura, T. Hiramatsu, K. Shimada, K. Matsui*, Kanto Gakuin University, Japan

Conducting polymer polythiophene (PT) films incorporated with highly-functional molecules such as copper phthalocyanine (CuPc), fullerene (C60) and rhodamine B (RB), further, tetrathiafulvalene (TTF) which is an donor and tetracyanoquinodimethane (TCNQ) which is an acceptor were synthesized and characterized by photoluminescence measurements (PL), time correlated single photon counting (TCSPC) life time measurements and fourier transform infrared spectroscopy (FTIR) in order to obtain fundamental photoluminescence properties of the polymer complexes. Those molecules were doped in the polymer film by the diffusion method. The solvents used in the doping process were acetonitrile or toluene. In the FTIR measurement, the molecular vibration mode of each molecule was observed in each polymer, and it was observed that each molecule had been doped in PT film. A photoluminescence single emission peak was observed at 610 nm in the case of PT doped sample with CuPc by diffusion method. Adding TCNQ molecules to the CuPc diffused PT sample by the diffusion method made the emission peak position varied and the photoluminescence intensity varied. A photoluminescence double emission peak was observed at 590nm and 738nm in the case of PT doped sample with C60 by diffusion method. Double emission peak was observed at 610nm and 663nm when TTF was added after CuPc doping. When TCNQ was added after CuPc doping, emission peak became a 480nm single peak. A photoluminescence double emission peak was observed at 590-660 nm in the case of PT doped sample with RB by diffusion method. In the case of using acetonitrile as a solvent, emission peaks were 610nm and 660nm. In the case of using toluene as a solvent, emission peaks were 590nm and 660nm. As for intensity, the low wavelength side became stronger. Adding TTF and TCNQ molecules to the RB diffused PT sample by the diffusion method made the emission peak position varied and the photoluminescence intensity varied. In the case of PT doped RB and TTF using toluene as a solvent, emission peak was a 590nm single peak. In the TCSPC measurements, 2 or 3 life time components with several tens of nsec to several hundreds of nsec existed in the case of highly-functional molecules doped PT. Adding TTF or TCNQ caused the life time change in the components. It suggests that the change of the emission states in the polymer complexes causes the shift of the emission peaks and the change in intensity.

This work was aided by MEXT-supported Program for the Strategic Research Foundation at Private Universities.

TF-ThP13 Production of Miniaturized Optical Interference Filters Array for CMOS Sensor. *C.-N. Hsiao, P.-K. Chiou, H.-P. Chen, B.-H. Liao, Y.-W. Lin, F.-Z. Chen*, Instrument Technology Research Center, Taiwan

Optical interference filters designed for use in a space-grade multispectral assembly in a complementary metal-oxide-semiconductor sensor were deposited on fused silica by ion-beam-assisted deposition. The optical parameters of optical interference coatings were optimized using admittance loci analysis. The patterned multispectral assembly containing blue, green, red, near infrared, and panchromatic multilayer high/low alternated dielectric band-pass filter arrays in a single chip was fabricated by photolithography process. The corresponding properties of the films were investigated by *in situ* optical monitoring, ellipsometry, spectrometry, scanning electron microscopy and high resolution scanning transmittance microscopy. It was found that the optical properties were significantly improved by employing ion-beam-assisted deposition. The average transmittances were above 90 % for the multispectral assembly, with a

rejection transmittance of less than 1% in the spectral range 350–1100 nm. To estimate the optical stability of optical coatings for aerospace applications, a space environment assuming a satellite orbiting the Earth at an altitude of near 780 kilometers was simulated by a Co^{60} gamma (γ) radiation test.

TF-ThP14 Analysis of Thin Layers with Low Energy Ion Scattering (LEIS). *B. Hagenhoff, M. Fartmann, D. Breitenstein*, Tascon GmbH, Germany, *T. Grehl, ION-TOF GmbH*, Germany, *H.R.J. ter Veen*, Tascon GmbH, Germany

When growing thin layers – through ALD or other processes – it is important to know how the layer is growing. Low Energy Ion Scattering (LEIS) can play a pivotal role in the study of film growth. It can be used for a quantitative analysis of the outermost atomic layer. This feature is used to determine layer closure or the existence of pinholes.

At the same time, LEIS gives information about the composition below the surface (similar to RBS). This so called "static depth profiling" can be used to monitor and study the evolution of layer thickness.

Examples will be shown for ALD Ta layers on Si, demonstrating the capabilities of LEIS to determine layer closure and the development of the layer thickness in the ALD process, also at low cycle numbers.

A Diamond Like Carbon (DLC) on Si system will be shown to demonstrate the possibilities to combine static depth profiling with dynamic (sputter) depth profiling, combining the advantages of both techniques and overcoming their drawbacks.

TF-ThP15 Reaction Mechanism for the Atomic Layer Deposition of Titanium Dioxide using Titanium Tetrachloride and Titanium Tetraisopropoxide as Precursors. *R.P. Chaukulkar, S. Agarwal*, Colorado School of Mines

Atomic layer deposition (ALD) is a thin film deposition technique widely used to deposit highly conformal thin films of a wide range of materials including metal oxides and nitrides, metals and more recently, polymers. Most ALD processes for the deposition of metal oxides require the use of H_2O , O_2 plasma, O_3 , or H_2O_2 as the oxygen source. ALD processes for depositing metal oxides using metal halides and metal alkoxides as the oxygen source were first reported by Ritala (Science **288**, 319 (2000)) to mitigate the problem of an interfacial oxide formation during deposition on semiconductor surfaces such as Si and Ge. Herein, we report an ALD process to deposit titanium dioxide using TiCl_4 and titanium tetraisopropoxide ($\text{Ti}[(\text{OC}_3\text{H}_7)_3]$, TTIP) as the oxygen source. We have used *in situ* attenuated total reflection Fourier transform infrared spectroscopy to probe the corresponding surface reactions during film growth over a temperature range of 150–250 °C. Depending on the surface temperature, alkyl-transfer and β -hydride elimination have been proposed as two possible reaction pathways for TTIP on a TiCl_4 -terminated surface. However, our infrared data show that alkyl-transfer is the only reaction pathway for this ALD process even at temperatures of up to 250 °C, which is close to the decomposition temperature of TTIP. We also report the growth per cycle, stoichiometry, and the band gap for these TiO_2 films grown over the above temperature range. Finally, we discuss the growth of these TiO_2 films on Cl-terminated Ge nanoparticle surfaces.

TF-ThP16 Nitrogen Doped Zinc Oxide Thin Films Prepared by Reactive RF Magnetron Sputtering of Zinc in Nitrous Oxide Atmosphere and Post-deposition Annealing Structural and Optical Properties. *L.A. Hernández-Hernández*, ESFM-IPN, Mexico, *A. Hernández-Hernández, F. De Moure-Flores, J.G. Quiñones-Galván*, CINVESTAV-IPN, Mexico, *J.J. Araiza-Ibarra, UAF-UAZ*, Mexico, *M. Meléndez-Lira*, CINVESTAV-IPN, Mexico

Nitrogen doped zinc oxide thin films were deposited on glass and silicon substrates by reactive magnetron RF sputtering of zinc in a N_2O -Ar ambient. The deposition conditions were optimized varying the substrate temperature and the $\text{N}_2\text{O}/\text{Ar}$ sputtering gas ratio. Representative films were studied employing structural, optical and spectroscopic techniques. A correlation between the nitrous oxide partial pressure, the chemical composition and the crystalline structure of the films was obtained. Stoichiometric and highly oriented ZnO thin films along the (0 0 2) crystallographic direction were obtained for a nitrous oxide partial pressure of ~33%. Lower temperatures produced samples with higher nitrogen content and slightly higher band-gap energy. Post-deposition annealing treatments in nitrous oxide atmosphere at 500 °C significantly improved the crystallinity of the samples as confirmed by x-ray diffraction.

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TF-ThP17 Physical and Electrical Characteristic of Atomic Layer Deposition of $\text{Al}_x\text{Hf}_y\text{O}_z$ on Silicon. *Y. Lin, W. Li, S. Fanz, R. Candler*, UCLA

In order to meet the increasing demand for high frequency electronic devices, the physical dimensions of MOSFETs have been continuously scaled down into nanoscale. However, one of the bottlenecks we encounter during the scaling-down process is the tunneling current leakage at the gates. SiO_2 , the most commonly used traditional gate dielectric experiences an appreciable amount of tunneling current when the gate thickness is below 1–1.2 nm. And leakage greatly degrades the performance of nanoelectronics. Therefore, we propose using high-k dielectrics to replace SiO_2 , which can effectively limit the tunneling leakage without losing the current control at gates. Our research has mainly focus on $\text{Al}_x\text{Hf}_y\text{O}_z$ deposited on silicon via Atomic Layer Deposition (ALD). The Aluminum to Hafnium ratio in the oxide is tuned to maximize the electrical and physical properties of the film. The electrical properties of each oxide will be characterized by fabricating transistors with gate oxide thicknesses of 5, 10, and 15 nm. Other than taking C-V and I-V measurements for capacitors and transistors, the films will be characterized by XPS, AFM, and spectroscopic ellipsometry. Finally, the effects of various annealing and deposition temperatures at the silicon-oxide interface will be studied using TEM.

TF-ThP18 Wetting Properties of Silicon Incorporated DLC Films. *T.G. Kim*, Pusan National University, Korea

Recently, Diamond-like carbon (DLC) films have come to the center stage of developing coatings for moisture resistant lubricant, water repellent and cathode for lithium batteries. Hydrophilic nature of DLC films played an important role in the above applications.

In this study, Silicon incorporated diamond-like carbon films were deposited on aluminum substrate by a radio frequency plasma-enhanced chemical vapor deposition method. The control of hydrophilic of Silicon incorporated diamond-like carbon surfaces has been studied by the use of O_2 plasma etching and heat treatment. The characteristic of DLC films was evaluated by various techniques such as Contact angle, Micro Raman spectroscopy and Nano indentation. Contact angle of Si-DLC film was about 60°. The contact angle was decreased into about 2° by not only by oxygen plasma treatment for 10min but by heat treatment at 700°C, respectively. In addition, increase of heat treatment temperature makes the contact angle of Si-DLC film decrease.

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