Thin Films
Room Ballroom BC - Session TF-ThP
Thin Film Poster Session

TF-ThP-1 Thickness and Elemental Quantification of (Ultra)Thin Films Revisited, ***Markus Sauer***, *Jakob Rath, Annette Foelske,* TU Wien / AIC, Austria; *Dieter Ingerle,* TU Wien / XRC, Austria

Many approaches have been taken towards precise determination of overlayer thickness and (elemental) quantification of thin/ultrathin films (0.5-100nm). X-ray reflectivity (XRR) and X-ray photoelectron spectroscopy (XPS) as well as spectroscopic ellipsometry are commonly used to provide information about sample composition and layer depth (1-3). However, each of these methods has its limitations and specific techniques/sample geometries etc. might require extensive preparation and or do not allow for the use of ultra-high vacuum instrumentation. In addition, some of these methods as well as alternatives like Rutherford Backscattering/Elastic Recoil Detection Analysis (RBS/ERDS) require expensive equipment and/or access to large-scale facilities which is not always an alternative in every day-use cases.

Herein we report a broad comparison of different techniques including most of the above-mentioned ones (XPS, SEM-EDX, XRR, Ellipsometry) as well as Auger-Meitner Electron Spectroscopy (AMES), X-ray fluorescence (WXRF and GIXRF) and Raman spectroscopy for two sets of reference materials: HfO_2 on SiO_2/Si (4) and Fe/Ni thin films with different relative compositions.

We provide an approach for choosing different methods and method combinations depending on the requirements/sample surface size/roughness etc. for laboratory scale application beyond the reference material case. In addition, limitations of each method in terms of precision and applicability are discussed.

A roadmap is laid out for finding the most useful way of reaching the desired precision for quantification and thickness determination trying to use methods that are available to a large number of researchers in academia and industry.

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(2)Donald R Baer, Yung-Cheng Wang, David G Castner, Use of XPS to Quantify Thickness of Coatings on Nanoparticles, *Microscopy Today*, Volume 24, Issue 2, 1 March 2016, Pages 40–45, doi: 10.1017/S1551929516000109 [https://doi.org/10.1017/S1551929516000109]

(3)S. Terada, H. Murakami and K. Nishihagi, "Thickness and density measurement for new materials with combined X-ray technique," *2001 IEEE/SEMI Advanced Semiconductor Manufacturing Conference (IEEE Cat. No.01CH37160)*, Munich, Germany, 2001, pp. 125-130, doi: 10.1109/ASMC.2001.925630.

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TF-ThP-2 High-Mobility of Amorphous-Crystalline Phase-Composite Indium Oxide for Thin Film Transistor Applications, ***Quang Khanh Nguyen,*** Hanyang University, Viet Nam; *Myung Mo Sung,* Hanyang University, Republic of Korea

Indium oxide (InO_x) exhibits high electron mobility and optical transparency, ideal for advanced thin-film transistors (TFTs). However, its performance is often constrained by excessive carrier concentration and challenges in crystallization control. In this study, an amorphous/nanocrystal phase-composite InO_x films are fabricated using high-pressure atomic layer deposition (ALD) with InCA-1 as the metal precursor and H_2O_2 as the oxidant. The amorphous matrix within the phase-composite InO_x promotes resonant hybridization, facilitating efficient electron transport by forming delocalized states through the overlap of nanocrystalline and amorphous wave functions. By systematically varying deposition temperature and channel thickness, we achieve precise control over carrier concentration and phase composition. The optimized InO_x thin films, deposited at a low deposition temperature of 110 °C with a 7.0 nm channel thickness, exhibit outstanding field-effect mobility (61.1$cm^2 V^{-1} s^{-1}$), high optical transparency, excellent surface coverage, remarkablemechanical flexibility, and strong environmental stability. This approach overcomes previous limitations in InO_x thin-film fabrication, broadening its potential for next-generation electronic applications.

TF-ThP-3 Electrical and Mechanical Stability of Flexible Low-Dielectric Constant Carbon-Doped Oxide (SiCOH) Thin Films Under Repeated Mechanical Stress, ***Rajib Chowdhury***, *SeonHee Jang,* University of Louisiana at Lafayette

The microelectronics industry continuously advances materials science to enhance integrated circuit (IC) performance. Interconnect structures are becoming critical as the transistor density increases. It also limits the chip speed due to increased resistance-capacitance (RC) delay. Traditionally, aluminum (Al) and silicon oxide (SiO_2) were utilized as metal and dielectric materials, which were replaced with copper (Cu) and low dielectric constant carbon-doped silicon oxide (low-*k* SiCOH, $k < 4$) to improve the RC delay and power consumption. Simultaneously, flexible electronics have gained attention, utilizing polymer substrates for applications like wearable devices and displays. However, integrating low-*k* flexible dielectric films with polymer-based substrates remains challenging due to the low glass transition temperatures of the substrates. Besides, it is essential to study the mechanical stability of materials for the integration of flexible electronic devices. This study explores the applicability of the low-*k* SiCOH thin films for flexible electronics by observing the effects of repeated mechanical bending tests.

Flexible low-*k* SiCOH films were produced onto flexible indium tin oxide-coated polyethylene naphthalate (ITO/PEN) substrates by plasma-enhanced chemical vapor deposition (PECVD) of a tetrakis (trimethylsilyloxy)silane precursor. The films were deposited at room temperature with the RF plasma power varied from 20 to 100 W. The films were subjected to bending tests with up to 10000 bending cycles. Mechanical characterization was performed by nanoindentation testing for the elastic modulus and hardness. Chemical bonds were characterized by Fourier transform infrared (FTIR) spectroscopy, and the atomic concentration was measured by X-ray photoelectron spectroscopy (XPS). The dielectric constant was measured from capacitance-voltage measurements.

The pristine SiCOH films had a mechanical strength of up to 9.1 GPa and a low *k*-value down to 2.00. The films were optically transparent, smooth, and hydrophobic. The prominent chemical peaks of CH_x, Si-CH_3, Si-O-Si, and Si-$(CH_3)_x$ were identified for pristine films from the analysis of FTIR spectra. Upon repeated mechanical bending tests with bending cycles up to 10,000, the flexible SiCOH films maintained their transparency, smoothness, and hydrophobicity and showed a stable *k*-value below 4.0. No significant changes in the FTIR spectra were observed, and no cracks or delamination were observed in the films. The SiCOH films showed stable physical, chemical, and electrical properties under repeated mechanical bending.

TF-ThP-4 The Impact of Copolymer Molecular Sequence on Electronic Transport, ***Mahya Mehregan***, *Jack Schultz,* University of Missouri-Columbia; *Matthew Maschman, Matthias Young,* University of Missouri, Columbia

This work demonstrates the successful formation of EDOT-co-Py copolymer thin films using oxidative molecular layer deposition (oMLD), with electrical conductivity values intermediate between those of PEDOT and PPy. By controlling the molecular sequence during copolymerization via the sequential surface reactions afforded by oMLD, we investigate the influence of monomer arrangement on electronic conductivity. Our findings reveal that the electrical conductivity of the copolymer thin films is not determined by the composition ratio of EDOT and Py but is instead strongly influenced by the block length of each monomer chain. These block lengths modulate the electron energy well depths for electron transport along the copolymer chains, which in turn affects conductivity. Our analysis reveals that the energy well depth in Boltzmann transport modeling exhibits a sigmoidal relationship with the separation distance between conductive domains, rather than the previously assumed linear dependence. We identified a critical domain size of >3 monomer units (corresponding to 1.4 nm) that significantly alters electronic conductivity, consistent with electron hopping distances observed in biomolecules. This suggests a universal length scale for electronic interactions in polymers.

TF-ThP-5 Synthesis of Bismuth-based EUV Photoresists using Molecular Layer Deposition, ***Jane Keth***, *Duncan Reece, David Bergsman,* University of Washington

Extreme ultraviolet (EUV) photolithography has seen substantial interest from the semiconductor industry as a tool to create sub-10 nm features, which are necessary to improve device performance. To use this process, EUV-compatible photoresists are needed that are highly absorbing of EUV light, can be deposited as a thin film, and have high etch resistance. Many photoresist materials have been explored to meet this need, including polymer films exposed to vapor-phase infiltrants, polymer films combined with metal additives, and hafnia-based nanoparticle thin films. However, these resists tend to be limited to low viscosity resist formulations or use deposition methods like spin coating that struggle to form conformal coatings. One promising strategy for creating these resists involves using molecular layer deposition (MLD) to synthesize hybrid inorganic-organic films directly on the surface of interest. MLD is a vapor-phase layer-by-layer thin film deposition process that can deposit films with subnanometer thickness and compositional control. While MLD has been used to make aluminum, hafnium, and tin-based EUV photoresists, films based on other elements may be beneficial. In this work, we will present on using a Bismuth-based MLD process to grow hybrid organic-inorganic EUV photoresists. Using a specialized parallelizing reactor unique to the Bergsman research group, we explore the growth of Bi-based photoresists with different organic linkers, characterizing their composition and testing their ambient stability and chemical stability. After studying the as-deposited resists, we treat the resists to UV light and measure their subsequent chemical structure and stability. This data is used to derive the understanding of how Bi-based EUV photoresists can be further optimized for EUV photolithography.

TF-ThP-6 Hollow-Cathode Plasma-Assisted ALD of CuO Thin Films: Evaluating Self-Limiting Growth Conditions and Material Properties*, Fatih Bayansal, Steven Allaby, Habeeb Mousa, Helena Silva,* ***Necmi Biyikli,*** University of Connecticut

Copper oxide (CuO) is a promising p-type semiconductor material with potential applications for energy and optoelectronic devices. In this study, we conducted a comprehensive saturation study within the scope of our initial attempts to grow CuO films by hollow-cathode plasma-assisted atomic layer deposition (HCP-ALD) followed by material characterization study to evaluate the structural, optical, and electrical properties of grown samples.

During CuO growth experiments, copper(II) hexafluoroacetylacetonate hydrate [Cu(hfac)$_2$·xH$_2$O] and O$_2$ plasma were used as the metal precursor and oxidizing agent, respectively. Saturation experiments performed on Si(100) substrates at 150 °C showed that the growth rate reached the saturation regime when the precursor pulse duration increased above a certain threshold. This demonstrated that surface-controlled self-limiting ALD behavior is achieved under appropriate plasma conditions. On the other hand, CuO formation was suppressed in the growths performed using only Ar plasma or O$_2$/Ar mixture, and metallic or non-stoichiometric structures were observed in some samples. These results confirmed the critical role of reactive oxygen species for CuO growth.

After determining the self-limiting growth window, the synthesis temperature was gradually increased to 250°C and film deposition studies were carried out on n-Si, sapphire, and quartz substrates. Initial transmittance measurements showed that as the temperature increased, the films exhibited higher transmittance in the visible region, thus increasing the film smoothness and quality.

X-ray diffraction (XRD) analyses revealed that the films grown in optimized O$_2$ plasma conditions contained polycrystalline CuO phases with (110), (002) and (111) planes. In Ar-oriented plasma environments, Cu$_3$N phases were observed, suggesting that oxidation was incomplete, or nitrogen doping occurred. These findings indicate that the HCP-ALD process is extremely sensitive to plasma composition and precursor-plasma interactions.

Hall effect, XPS, and TEM analyses are ongoing to determine the electrical, chemical, and structural properties. This research provides the basis for reliable CuO film growth at low temperature, and future process optimizations are aimed at the production of phase-pure, stoichiometric, and electrically active p-type CuO films.

TF-ThP-7 Low-Temperature Atomic Layer Deposition of ZnO Thin Films on Cotton for Flexible Electronics*, Habeeb Mousa,* ***Steven Allaby****, Fatih Bayansal,* University of Connecticut*; Md Sazid Bin Sadeque, Tamer Uyar,* Cornell University*; Helena Silva, Necmi Biyikli,* University of Connecticut

The development of flexible electronics has advanced rapidly, with applications from sensors and energy storage to wearables. Among these, photodetectors (PDs) are of growing interest due to their potential roles in health monitoring, security, and optical communication. Zinc Oxide (ZnO), with its wide bandgap, stability under long-term light exposure, and high sensitivity to UV/visible radiation is an ideal material for such devices. However, fabricating thin film-based devices on textiles often affects their mechanical properties such as flexibility, durability, and washability. This work presents an approach that leverages low-temperature atomic layer deposition (ALD) of ZnO on cotton to achieve flexible PDs while preserving the inherent properties of cotton.

ZnO was deposited on cotton (woven bleached, 98 gsm) substrates using diethylzinc (DEZ) and H2O as Zn precursor and co-reactant respectively in a thermal ALD reactor at 120 °C. The unit ALD cycle in which 20 sccm N2 is used as the carrier gas consists of 0.5s DEZ pulse, 30s purge, 0.5s H2O pulse, 30s purge steps. Following the deposition of ZnO layers on cotton, interdigitated electrodes consisting of 200 nm Cr was evaporated by e-beam deposition to create the metal-semiconductor-metal (MSM) structures.

The resulting ZnO films on cotton are characterized in terms of their structural, morphological, compositional, and photo-response properties. X-ray diffraction analysis revealed the polycrystalline nature of the as-grown ZnO layer on cotton. The photo-response characteristics of the fabricated MSM-PD device structures were placed under a solar simulator (Newport 94022A) at a distance of ~20 cm. The bias voltage was scanned from –10 to 10V in a 100-mV step under dark and illuminated conditions. The resulting photo-current at 10V bias showed ~160-fold increase when compared to dark current (from 5.5 nA to 888 nA). Moreover, our study displays an effective ZnO-based photodetector on cotton at low bias voltage of 1V where the photocurrent increased from 0.58 nA to 74 nA (~128 fold increase) highlighting the potential for low-power wearable sensing applications. In order to investigate the sensitivity and stability of the device, the photocurrent—time measurements were conducted by applying five 'ON/OFF' pulses at a bias voltages 10 V. The 'ON' state and 'OFF' state lasted for 5 mins each. The sensitivity was calculated and found to be 271. Future studies could focus on further characterizing the spectral photo-response under various environmental conditions and optimizing the device architecture by exploring different doping strategies, or composite structures that can enhance light absorption.

TF-ThP-8 The Impact of Bismuth Surfactants on MBE-Grown Insb Thin Films for Applications in Mid-Infrared Devices*,* ***Pan Menasuta,*** 309 Boston Ave*; John H. McElearney, Thomas E. Vandervelde,* Tufts University

Indium antimonide (InSb), an important narrow direct bandgap semiconductor (0.17 eV at 300K), is highly optically sensitive in the mid-wave infrared (MWIR, 2-5 µm) spectrum. InSb-based devices are crucial for thermal imaging, spectroscopy, and astronomy as a result of atmospheric transmission and thermal emission characteristics. [1–5]. Its broad sensitivity (1.5-7 µm) also enables gas detection. However, reproducible growth of high-quality InSb epitaxial layers via molecular beam epitaxy (MBE) is challenging due to its low melting point. The epitaxial process requires lower growth temperatures and is prone to crystalline and surface defects. Optimal InSb growth occurs at 385°C with a V: III ratio of 1.2. Accurate temperature control is challenging at these lower temperatures, which further complicates the narrow optimal growth range and can negatively impact the film properties. Controlling surface morphology during growth is critical for advanced optoelectronic devices.

Bismuth surfactancy in MBE has been shown to improve surface morphologies in many III-V materials [6, 7]. A very low bismuth flux can modify the adlayer surface before desorption, and has been shown to improve the morphology of the surface in multiple materials [6–8].To our knowledge, no systematic studies have been reported on the effects of Bi surfactancy on MBE growth of InSb thin films [6, 7, 9]. This study investigates the effects of Bi surfactancy on InSb MBE growth over a wide range of growth temperatures (280-410°C). Two series of homoepitaxial InSb(100) films were grown by MBE: a control set and a set grown with Bi surfactancy, with identical parameters otherwise. The temperature was calibrated using the RHEED pattern transition c(4x4) to a(1x3), which is reported to occur at 370°C [10, 11]. The surface morphology and elemental distribution were analyzed using AFM and SEM-EDS, while XPS confirmed the absence of Bi incorporation. Finally, TEM was performed to analyze the film's lattice structure.

TF-ThP-9 Strategically Introducing Interfaces into Refractory Concentrated Alloys to Increase Tolerance in Extreme Environments*,* ***Benjamin Derby****, Yao Li,* Los Alamos National Laboratory

This work introduces three-dimensional interfaces into refractory concentrated alloys to increase mechanical and conductivity performance in extreme environments. Physical vapor co-deposition at elevated temperature kinetically forces the system to phase separate into alloy architectures with unique 3D interface structures. The increase in dynamic strength and deformability of these structures are tested using a novel high-strain-rate nanoindenter. These materials provide a rapid prototyping framework for developing bulk materials with optimized performance in complex, extreme environments.

TF-ThP-10 Raman Scattering as a Probe for Tuning Magnetic Quasiparticles in NiO Thin Films Through Ion Beam Irradiation*, Simranjeet Kaur,* *Indian Institute of Technology Delhi, India*

NiO is a wide-band transparent insulator, exhibiting a bandgap of 3.6 eV-4.0 eV [1]. It is an antiferromagnetic material with a Néel temperature of 523 K. NiO crystallizes in a NaCl-type face-centered cubic structure with a lattice parameter of 0.417 nm. The antiferromagnetic order in NiO is due to the antiferromagnetic alignment of ferromagnetic (111) planes along the [111] crystallographic direction[2].Below T_N,magnetic ordering induces a rhombohedral distortion.This study presents the growth of (111)-oriented NiO thin films on (0001)-sapphire substrate using pulsed laser deposition (PLD). DC magnetic susceptibility measurements of the films confirm that they maintain antiferromagnetic ordering at room temperature. Additionally, this finding is supported by the observation of two-magnon(2M) Raman scattering.The relative intensity of this 2M mode compared to a neighboring phonon mode further highlights the bulk-like antiferromagnetic state in the thin films. NiO thin films were irradiated utilizing an Au ion beam at varying fluences.X-ray diffraction (XRD) analysis indicated a broadening and a shift towards a higher 2θ value in the NiO(111) peak with increased fluence, which suggests a reduction in the out-of-plane lattice parameters.Atomic force microscopy (AFM) results demonstrated increased surface roughness of the films post-irradiation.DC magnetic susceptibility measurements showed a decrease in the magnetic moment and Néel temperature at the higher fluence of $5x10^{12}$ ions/cm^2, attributable to defects induced by the ion beam irradiation. Raman spectroscopy further supported these findings, with significant changes observed in the two magnon peaks,which experienced a redshift and broadening in the irradiated samples. This shift and broadening signify a reduced antiferromagnetic coupling due to high-energy ion beam irradiation.Additionally,the 1P peak at 575 cm^{-1} exhibited a redshift and broadening in the irradiated samples. The ratio of I_{1P}/I_{2P} increased significantly upon irradiation and even surpassed one at the higher fluence value, indicating a higher degree of disorder induced by the ion beams. Overall, this study demonstrates the successful deposition of (111)-oriented NiO thin films via PLD, exhibiting magnetic properties similar to bulk NiO, and the tuning of the 2M peak by ion beam irradiation.These findings highlight the potential of NiO thin films for exploring fundamental magnetic interactions and developing optoelectronic applications.

[1]H. Ohta et al., Thin Solid Films 445, 317(2003)

[2]S. M. Rezende et al., J. Appl. Phys. 126, 151101(2019)

TF-ThP-11 Mesoporous Metal Fluoride Films with Ultra-low Tunable Refractive Index for Broadband Antireflection*, Choon-Gi Choi,* *Ki-Seok An,* Thin Film Materials Research Center, Korea Research Institute of Chemical Technology (KRICT), Republic of Korea

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Porous materials are of great interest in various fields such as optics, biology, energy, and catalysis. While energy and catalysis applications focus on achieving high porosity, optical applications demand not only low-refractive-index (RI) materials that overcome the limitations of naturally occurring substances but also the formation of a smooth RI gradient from the substrate to air. This requires both high porosity and precise control over it. Conventional methods for fabricating porous structures, including templating, self-assembly, and zeolitic synthesis, typically rely on sacrificial templates, which must be removed through chemical etching or thermal treatment, potentially damaging the host material and limiting scalability.

In this study, we present mesoporous metal fluoride films composed of MgF_2 and LaF_3, fabricated using a simple, template-free, one-step precursor-derived method. Pores spontaneously form during solidification due to the inherent instability of $La(CF_3OO)_3$. Electrostatic interactions between $Mg(CF_3OO)_2$ and $La(CF_3OO)_3$ precursors enable the controlled formation of mesoporous structures with finely tunable RI values ranging from 1.37 to 1.16. By stacking layers of $MgF_2(1–x)–LaF_3(x)$ with different compositions, a graded refractive index (GRIN) antireflection coating (ARC) is achieved, delivering excellent broadband performance with an average transmittance of ~98.03% in the 400–1100 nm range. Despite these advances, a refractive index gap still remains between the fluoride composite and air, primarily due to the inherently high RI of LaF_3.

To address this, we propose an innovative approach that enables precise tuning of porosity using micelle-assisted MgF_2 precursor intermediates. As micellization increases the size of the MgF_2 precursor clusters, the resulting solidified MgF_2 grains and the intergranular voids between them also increase, allowing for the fabrication of MgF_2 structures with ultra-low RI (~1.04) and fine RI control increments. When applied as a GRIN ARC on quartz substrates, this strategy achieves an average transmittance of ~97.96% across the 250–1100 nm spectral range.

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