

## Fundamentals and Technology of Multifunctional Materials and Devices

### Room Sunrise - Session C1

#### Optical Metrology in Design, Optimization, and Production of Multifunctional Materials

**Moderators:** Nikolas Podraza, University of Toledo, Juan Antonio Zapien, City University of Hong Kong

**1:50pm C1-2 Design Principles for Binary and Multicomponent Conductive Nitrides for Applications in Electronics Plasmonics and Photonics, Panos Ntiras, Aristotle University of Thessaloniki, Greece; N Kalfagiannis, Nottingham Trent University, UK; S Kassavetis, Aristotle University of Thessaloniki, Greece; G Abadias, Université de Poitiers, France**

Although conductive nitrides, such as TiN, ZrN, and  $Ti_xAl_{1-x}N$ , were intensively studied for mechanical applications since 1980s and as diffusion barriers since 1990s, their combination of thermal and mechanical stability, with the compatibility of their growth to CMOS fabrication, and with their refractive character and electronic conductivity, paved the way for their emergence as important plasmonic and photonic materials [Naik, G.V. *et al*, Adv. Mater. 25 (2013) 3264-3294; Kassavetis, S. *et al*, Surf. Coat. Technol. 295 (2015) 125-129; Kassavetis *et al*, Appl. Phys. Lett. 108 (2016) art. No. 263110; Metaxa *et al*, ACS Appl. Mater. Inter. 9 (2017) 10825-10834]. In this work, we review the optical properties, in terms of ellipsometry, FTIR spectroscopy, and XPS valence band spectra, of a wide range of binary (TiN, ZrN, HfN, VN, NbN, TaN, MoN, WN) and multicomponent ( $Ti_xMg_{1-x}N$ ,  $Ti_xSc_{1-x}N$ ,  $Ti_xAl_{1-x}N$ ,  $Ti_xZr_{1-x}N$ ,  $Ti_xHf_{1-x}N$ ,  $Ti_xNb_{1-x}N$ ,  $Ti_xTa_{1-x}N$ ,  $Ti_xMo_{1-x}N$ ,  $Ta_xZr_{1-x}N$ ) films and we establish correlations between their optical behavior, electron conductivity and work function with the intrinsic (*e.g.* the valence electron configuration of the constituent metal) and extrinsic (*e.g.* point defects and microstructure) factors. We also correlate the plasmonic performance of nitride nanostructures and nitride/dielectric interfaces with the electron density of states of their valence band. We demonstrate that, indeed TiN and ZrN along with HfN are the most well-performing plasmonic materials in the visible range, while VN and NbN may be viable alternatives for plasmonic devices in the blue, violet and near UV ranges, albeit in expense of increased electronic loss. WN is disregarded as candidates for plasmonics, opposing recent theoretical works, due to the excessive concentration of point defects, even in epitaxial form. Finally, TaN has a substantial plasmonic activity in the metastable, cubic rocksalt structure, however, in most cases tends to form mixed cubic-hexagonal samples that are also excessively lossy. Furthermore, we consider the alloyed ternary conductive nitrides and by critical evaluation and comparison, we identify the emerging optimal tunable plasmonic conductors among the immense number of alloying combinations. As a result, we provide design principles of nitride conductors for plasmonic, photonic, and optoelectronic devices, such a nanoantennas, SERS-based biosensors, selective absorbers of solar or other radiation, stable Bragg-mirrors, epsilon-near-zero (ENZ) metamaterials, and ohmic contacts for light emitting diodes based on III-V semiconductors.

**2:10pm C1-3 Tip Enhanced Optical Microscopy and Spectroscopy Based on Near Field Force Detection – a Review, H. Kumar Wickramasinghe, University of California, Irvine, USA**

**INVITED**

Near field scanning optical microscopy (NSOM) has evolved into a rich field of study with many different variants over the past 25 years. Many different modes of NSOM based on apertureless/scattering NSOM techniques have evolved such as near-field fluorescence and Tip Enhanced Raman Spectroscopy (TERS) etc. All these techniques are based on measuring a local tip enhanced near-field interaction in the far field. In this talk we present a review of a new modality where optical microscopy/spectroscopy is performed by measuring the dipole-dipole interaction force between an optically driven sample and a dipole created in the tip – here, a near field optical interaction is measured *in the near-field*. Photo induced force microscopy (PIFM) is capable of measuring both the linear and non-linear optical response of a sample on the nanoscale. We will present recent experimental and theoretical data both in the visible and in the mid infra-red.

**2:50pm C1-5 Crystallite Grain Orientation Manipulation through Deposition Flux Angle and Composition in  $CdSe_{1-x}Te_x$ , Dipendra Adhikari, M Junda, C Grice, P Koirala, Y Yan, R Collins, N Podraza, University of Toledo, USA**

Cadmium telluride (CdTe) based semiconductors are of interest as absorber layers for thin film photovoltaics. In particular, alloying with selenium ( $CdSe_{1-x}Te_x$ ) has helped to significantly improve device efficiency by reducing parasitic absorption losses in the vicinity of the n-type heterojunction partner. Here microstructural properties of two series of films are studied using grazing incidence x-ray diffraction measurements (GIXRD), scanning electron microscopy, and spectroscopic ellipsometry. A CdTe series consists of films deposited by sputtering onto soda lime glass substrates mounted at 0°, 45°, 55°, 65°, 75°, and 85° source flux angles relative to the substrate normal (*i.e.* glancing angle deposition). A  $CdSe_{1-x}Te_x$  series is fabricated by co-sputtering CdSe and CdTe with varying combinations of individual cathode powers resulting in a film series that spans the full range of compositions from  $x = 0$  to 1. Influence of deposition angle and film composition on resultant crystalline grain size and orientation are tracked for these films. All CdTe films studied are found to have cubic crystal structure and (111) preferential grain orientation. Films deposited at 0° and 45° are almost entirely (111) oriented, whereas films deposited at intermediate angles exhibit a wider variety of competing grain orientations, suggesting that deposition angle can be used as an effective parameter towards controlling grain orientation. With increasing numbers of grain orientations, grain size is found to decrease.  $CdSe_{1-x}Te_x$  alloys exhibit diffraction peaks corresponding to both cubic and hexagonal crystal systems. The films have a (111) preferred grain orientation that shifts from lower to higher values of  $2\theta$  with increasing Se content. These  $CdSe_{1-x}Te_x$  films are measured in both as-deposited and CdCl<sub>2</sub> treated states with all CdCl<sub>2</sub>-treated samples having increased grain size compared to corresponding as-deposited samples. Generally, the diffraction patterns transition from CdSe-like to CdTe-like with increasing  $x$ . However, interesting behavior is observed for intermediate compositions, such as the (103) diffraction peak corresponding to hexagonal crystal system becoming relatively strong for a few compositions with low  $x$ , but being weak for all others.

**3:10pm C1-6 Durable Electrochromic Coating Systems for Advanced Smart Windows and Security Devices, F Blanchard, B Baloukas, S Loquai, J Klemberg-Sapieha, Ludvik Martinu, Polytechnique Montréal, Canada**

The present work is our latest contribution to the development of large area smart windows based on electrochromic materials that have yet to properly breach the market due to the following main limitations: 1) the fabrication costs are still quite high, and 2) the durability of the system as a whole still requires significant improvements.

In response, this study offers a highly attractive and counterintuitive solution to both of these issues. Traditional WO<sub>3</sub> films, the main constituent of electrochromic windows, are traditionally deposited by magnetron sputtering at relatively high pressures (10-30 mTorr) to generate sufficient porosity and thus ensure a high ionic mobility. In this work, we explore a new and different approach to control the porosity involving intense ion bombardment during deposition at low pressures (< 5 mTorr). The resulting films' performance is tested through cyclic voltammetry using both H<sup>+</sup> and Li<sup>+</sup> ions and other complementary methods. We systematically categorize the coating properties based on their coloration efficiency, dynamic behavior and chemical durability. The present ion bombardment approach leads to a deposition rate increase of five times compared to the standard fabrication method, while the long term stability is significantly enhanced. The film characteristics are explained in terms of a microstructural model based on the formation of a unique nanocrystalline porous structure. In combination with an appropriate control of the surface reactions, this offers a possibility to tailor the transmission and reflection spectra of such coatings with enhanced durability for various applications such as advanced glazings for architectural glass, color shifting active security and authentication devices, and others.

**3:30pm C1-7 From “n” and “k” to Solar Cell Functionality: The Importance of Optical Property Characterization, Nikolas Podraza, M Junda, I Subedi, K Ghimire, University of Toledo, USA**

Predominate types of photovoltaic (PV) technologies studied at present were invented in the last century. Industrially manufactured devices based on wafer silicon (Si) and thin film cadmium telluride (CdTe) still have relevant problems with respect to their characterization. Methylammonium lead iodide perovskite (CH<sub>3</sub>NH<sub>3</sub>PbI<sub>3</sub>) materials have recently achieved very high efficiency when implemented as PV absorber

# Tuesday Afternoon, April 24, 2018

layers. Here the optical properties of layers in each of these types of devices will be discussed. For “past-to-present” generation materials, Si wafer based PV with aluminum (Al) back surface fields (BSF) are modeled with particular attention paid to the characterization of Al+Si interfacial region optical properties as obtained by through-the-Si spectroscopic ellipsometry. When Al+Si optical properties are obtained and incorporated into the ray-tracing simulation of Si wafer solar cell modules, good convergence is obtained between those simulations and experimental results also with device performance parameters such as short circuit current density ( $J_{sc}$ ) from quantum efficiency simulations (QE) aligned with experimental results. Next, “present” generation thin film PV including CdTe device structures are characterized by through-the-glass spectroscopic ellipsometry and modeled with QE based on reference optical properties to illuminate sources of optical and electrical losses. When CdTe is alloyed with selenium (Se), the band gap is narrowed and device performance is altered both in simulation in experimental QE results. Similarly, “future” generation CH<sub>3</sub>NH<sub>3</sub>PbI<sub>3</sub> perovskite based solar cells are characterized by through-the-glass ellipsometry with QE modeling matching experiment. Band gap narrowing of this class of perovskites is characterized with spectroscopic ellipsometry. The resulting optical constants are used in the simulation of perovskite based single junction and tandem junction solar cells.

3:50pm **C1-8 Bipolar Resistive Switching Performance of MoS<sub>2</sub> Based ReRAM Devices using WN as Bottom Electrode for Non-volatile Memory Application**, *Ravi Prakash, S Sharma, D Kaur*, Indian Institute of Technology Roorkee, India

The reproducible resistive switching characteristics of sputtered deposited MoS<sub>2</sub> thin film has been investigated in Cu/MoS<sub>2</sub>/WN stack configuration for resistive random access memory (ReRAM) application. Excellent bipolar resistive switching (RS) properties have been observed at a low voltage of +2.1 V and -2.5 V respectively, which favors device to reduce the power consumption. The advantages of employing WN over Pt or Ti as bottom electrode material were demonstrated such as the low resistive state value and uniformity of other RS parameters like endurance and retention. Formation/disruption of the conducting filament is verified as the main cause of exhibiting the RS properties. Ohmic behavior and trap-controlled space charge limited current (SCLC) conduction mechanisms are confirmed as dominant conduction mechanism at low resistance state (LRS) and high resistance state (HRS). High resistance ratio ( $10^2$ ) corresponding to HRS and LRS, good write/erase endurance ( $10^3$ ) and non-volatile long retention ( $10^3$  sec) are also observed. This study demonstrated that the MoS<sub>2</sub> thin films with WN bottom electrode have a great potential for future non-volatile ReRAM application.

## Fundamentals and Technology of Multifunctional Materials and Devices

Room Sunrise - Session C3

### Thin Films for Energy-related Applications

Moderator: Per Eklund, Linköpings Universitet

8:00am **C3-1 Synthesis and Optical Characterization of Cds Thin Film Obtained by Colloidal Technique**, *Laura Reyes, C Villa, R Villa, B Valdez, D Mateos, M Curiel, S Romero*, Instituto de Ingeniería, Universidad Autónoma de Baja California, Mexico

On the way to find a simple and economic technique that satisfy the requirements for further photonics applications; it was found out that the polymer-semiconductor nanocompound is a new generation of hybrid organic-inorganic materials. In this case, the inorganic semiconductors are synthesized in a polymeric matrix (organic). This kind of compounds has a huge interest due to its optical, electric, mechanical, thermal and magnetic properties comparing with other materials.

Semiconductors from groups II-VI such as CdSe, CdS, ZnS, PbS, among others, can be synthesized with polymers. In order to produce nano-polymer compounds, easily to remove if it is necessary, have great applications in optoelectronics devices. CdS meets these characteristics and can be used in photovoltaic (PV) cells, electronic and optoelectronic devices, e.g., due to the band gap of 2.42 eV.

In the present work CdS colloidal spheres were synthesized by using the solvothermal route technique, with the reaction given between a polymeric matrix (PVP), a Cd/Te salt and thiourea TU in a buffer alkaline solution technique. This allows the control of particle size and distribution to obtain: (i) thin, (ii) homogenous, (iii) strong adhesion and (iv) transparent films. The CdS colloidal spheres physical and optical properties were characterized by using FTIR spectroscopy, SEM-EDS and UV-VIS to compare the theoretical - practical capacity of absorbing photons in a different emission.

FTIR measurements show two characteristic peaks at  $600\text{ cm}^{-1}$  which correspond to the Cd-S stretching vibrational modes and  $1100\text{ cm}^{-1}$  which correspond to presence of S. UV-VIS absorption spectrum measured correspond to CdS. EDS analysis reveals the presence of S and Cd in the deposited thin films.

A colloidal solution was prepared and multi-deposited on soda lime glass covered with ITO substrate by spin-coating technique and annealed at  $120^\circ\text{C}$ . Ellipsometry determined the transparency and film thicknesses.

The results shown that nanostructures can be deposited as thin films by colloidal technique at low-cost. This is attractive for optoelectronic applications as photonic absorbers as PV cells.

8:20am **C3-2 Electrochemical Characteristics of Ni<sub>x</sub>N Thin Films Deposited by DC and HiPIMS Reactive Magnetron Sputtering**, *J Keraudy, L Athouel, J Hamon*, IMN - Nantes, France; *B Girault, D Gloaguen*, GeM - Saint-Nazaire, France; *M Richard-Plouet*, IMN - Nantes, France; *Pierre-Yves Jouan*, Université de Nantes, CNRS, France

Ni<sub>x</sub>N thin films can crystallize in many phases: Ni<sub>4</sub>N crystallizes in cubic phase, Ni<sub>3</sub>N in hexagonal phase and Ni<sub>2</sub>N in tetragonal phase. Most studies, related to NiN by reactive sputtering, report a mixture of nitrided phases and the change from Ni<sub>4</sub>N to Ni<sub>3</sub>N can be highlighted by the change in the magnetic behavior from ferromagnetic to paramagnetic. This material has great interest as negative electrode for Li battery and super-capacitors.

This study deals with optimization of DC and HiPIMS reactive magnetron deposition process using a pure nickel target (99.995%) in an Ar-N<sub>2</sub> gas mixture with varied nitrogen gas flow and bias voltage (floating or -100V).

The characterization of the NiN films has been carried out by X-ray diffraction (XRD), X-ray photoelectrons spectroscopy (XPS), Energy dispersive X-ray Spectroscopy (EDXS), SEM and AFM.

XRD measurements have highlighted the deformation of the Ni cubic cell as a function of nitrogen content and a mixture of nitrided phases (Ni<sub>4</sub>N, Ni<sub>3</sub>N and Ni<sub>2</sub>N) appears for 20% N<sub>2</sub> in the discharge.

XPS and EDX are well correlated and permit us to determine three zones: metallic between 0 and 20% N<sub>2</sub>, Ni<sub>4</sub>N between 20% and 42% N<sub>2</sub> and finally Ni<sub>3</sub>N for higher than 42% N<sub>2</sub>. These three zones are in good agreement not only with deposition rate and optical emission spectroscopy measurements but also with roughness, electrical resistivity and hardness.

Electrochemical characterizations, as cyclic voltametry, galvanostatic charge-discharge measurements and electrochemical impedance spectroscopy, have been performed in a conventional three-electrode cell using different aqueous electrolytes. The electrochemical behavior showing reversible faradaic peaks leads to the conclusion that the Ni<sub>x</sub>N film presents a charge storage mechanism of a so considered battery-type electrode, which is dependent of the amount of nickel implies in nickel-nitrogen bonds.

8:40am **C3-3 Photovoltaic Properties of Cu<sub>2</sub>O-based Heterojunction Solar Cells using n-type Oxide Thin Films Prepared by Magnetron Sputtering System with Loading Chamber**, *K Watanabe, H Tokunaga, Toshihiro Miyata, T Minami*, Kanazawa Institute of Technology, Japan

We recently reported that successfully obtained an excellent photovoltaic properties in p-n heterojunction solar cells fabricated by depositing appropriate n-type oxide semiconductor thin films using a pulsed laser deposition (PLD) method on p-type Cu<sub>2</sub>O sheets. However, the PLD method is not suitable for practical use because the deposition rate is low and it is difficult to prepare a n-type oxide semiconductor thin film with a large area. Although the magnetron sputtering method is suitable for practical use, excessive oxidation of Cu<sub>2</sub>O sheet surface due to oxygen plasma occurs. In this paper, we describe the photovoltaic properties for Cu<sub>2</sub>O-based heterojunction solar cells using the various n-type oxide semiconductor thin films prepared by the newly developed magnetron sputtering system with loading chamber. The magnetron sputtering apparatus have loading and deposition chambers, and used a d.c. and an r.f.(13.56 MHz) power supply that was applied either separately or together. One example, pre-sputtering procedure was carried out in a deposition chamber before introducing the p-Cu<sub>2</sub>O sheet from the loading chamber. After pre-sputtering, a p-Cu<sub>2</sub>O sheet was introduced into the deposition chamber, and then the n+-AZO thin films were prepared at RT at a pure Ar pressure of 0.6 Pa. Photovoltaic properties of n+-AZO thin film/p-Cu<sub>2</sub>O heterojunction solar cells was measured under AM1.5G solar illumination. A drastic improvement of the J-V characteristic was obtained by the pre-sputtering time was increased up to about 10 min. The J-V characteristics of the n+-AZO thin film/p-Cu<sub>2</sub>O heterojunction solar cells, prepared using the sputtering method with 10 min pre-sputtering, exhibited better properties than the PLD method. In addition, the solar cell's leakage current prepared by magnetron sputtering with 10 min pre-sputtering, measured under a reversed bias, was as low as the solar cell prepared by PLD. These results suggest that amount of oxygen supplied from the moisture adsorbed on the target surface was decreased by about 10min pre-sputtering process. As a result, the excessive oxidation of p-Cu<sub>2</sub>O sheets surface was suppressed. In addition, we also prepared AZO/n-type ZnO/p-type Cu<sub>2</sub>O solar cells. The photovoltaic characteristics of the solar cell with the AZO/n-type ZnO/p-type Cu<sub>2</sub>O structure improved compared with that of the solar cell with the AZO/p-type Cu<sub>2</sub>O structure. Therefore, the magnetron sputtering system with loading chamber is promising as a technology for preparing an n-type semiconductor thin film by suppressing excessive oxidation of the Cu<sub>2</sub>O sheet surface.

9:00am **C3-4 Synthesis of Tungsten Bronze by a Solution-based Chemical Route and the Near-Infrared Shielding Properties of Tungsten Bronze Thin Films**, *Pin-Jhen Wu*, National Cheng Kung University, Taiwan; *H Lu*, National Chin-Yi University of Technology, Taiwan; *S Brahma, J Huang*, National Cheng Kung University, Taiwan

In this study, we report the synthesis of Cs<sub>x</sub>WO<sub>3</sub> powders by a solution-based chemical route. The precursors are H<sub>2</sub>WO<sub>4</sub> and CsOH·H<sub>2</sub>O, and the solution is deionized water with triethylamine. The experiment can be done within a relatively short time. The as-synthesized and after heat-treatment Cs<sub>x</sub>WO<sub>3</sub> samples are characterized by X-ray diffraction, scanning electron microscopy, differential thermal and thermogravimetric analysis and fourier transform infrared spectroscopy. Next, the Cs<sub>x</sub>WO<sub>3</sub> powders are sintered at a fixed temperature for different time and Cs<sub>x</sub>WO<sub>3</sub> thin films are deposited by electron beam evaporation from the optimal sintered target. Then, the Cs<sub>x</sub>WO<sub>3</sub> films are annealed at different temperatures under Ar and H<sub>2</sub> atmosphere. The effects of annealing on the microstructure, morphology and NIR shielding properties of the Cs<sub>x</sub>WO<sub>3</sub> films are discussed.

9:20am **C3-5 ZnO Nano-structures Growth and Investigation**, *Alexander Azelevitch, I Lapsker*, Holon Institute of Technology (HIT), Israel

One of the main problems in the energy production field is limitation of usual fossil fuel sources. At the same time, traditional energy production technologies significantly influence on the global climate and the ecological state of our environment. This is the very important reason to find novel renewable and ecologically pure alternative energy sources. One of such

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sources is utilization of the piezoelectric effect for energy generation, harvesting and transfer.

Various piezoelectric materials are widely investigated in recent years. Zinc oxide (ZnO) is one of those materials. Thin films of ZnO have attracted great attention due to their unique piezoelectric and piezooptic properties, making them suitable for various microelectronics and optoelectronics applications, such as surface acoustic wave devices, optical fibers, solar cells etc.

In our work, we investigated possibility to grow ZnO nanostructures using thermal oxidation of a metal Zn coating prepared by vacuum evaporation on the rigid and flexible substrates. We used the glass, siall and polyimide substrates. Follows figure illustrates obtained nanostructure thin film. The structure and electrical properties of grown systems were studied. Also, it was shown application of the grown structures as the acoustic signal sensors.

9:40am **C3-6 Nitrogen Doping of ZnO Films by Decomposition of NO Gas using Heated Ir Wire in Catalytic Reaction-assisted CVD**, *Y Adachi, S Ono, A Kato*, Nagaoka University of Technology, Japan; *A Hashim*, MJIIT, Universiti Teknologi Malaysia, Malaysia; *Kanji Yasui*, Nagaoka University of Technology, Japan

The large bandgap (3.37 eV) and exciton binding energy (60 meV) of ZnO<sup>1)</sup> have recently stimulated intensive research into optoelectronic device applications, such as light-emitting diodes and laser diodes in the ultraviolet region. We previously developed a new chemical vapor deposition (CVD) method for ZnO film growth using a catalytic reaction over Pt-nanoparticles between dimethylzinc and high-temperature H<sub>2</sub>O. ZnO films grown on a-plane (11-20) sapphire (a-Al<sub>2</sub>O<sub>3</sub>) substrates exhibited excellent optical and electronic properties.<sup>2)</sup> In the present study, we attempted nitrogen doping of ZnO films by decomposition of NO gas using a heated Ir wire during film growth. The CVD apparatus and basic growth procedure have been previously reported, but without the use of the Ir wire. Epitaxial ZnO films were grown directly on a-Al<sub>2</sub>O<sub>3</sub> substrates at a substrate temperature of 773 K for 60 min without a buffer layer. The NO gas pressure was varied in the range 4.4'10<sup>-3</sup> to 1.3'10<sup>-1</sup> Pa. Although the residual carrier concentration was reduced by the addition of the NO gas, the ZnO films did not become p-type. In X-ray photoelectron spectra, multiple overlapping N-1s peaks were observed from 395 to 406 eV. By deconvolution of the spectra, components such as Zn-N, N-N, N-O, and NO<sub>x</sub> were identified. The relative intensity of the Zn-N peak at 395.5-361.1 eV increased when the heated Ir wire was used to decompose the NO gas.

Acknowledgement

This work was supported in part by a Grant-in-Aid for Scientific Research (No. 16H03869) from the Japan Society for the Promotion of Science.

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2) K. Yasui et al., MRS Symp. Proc., **1315** (2011) 21.

10:00am **C3-7 Morphology-Controlled Growth of ZnO Nanorods by Chemical Bath Deposition and Seed Layer Dependence on Their Structural and Optical Properties**, *Tomoaki Terasako, S Obara, S Sakaya, M Tanaka, R Fukuoka*, Ehime University, Japan; *M Yagi*, National Institute of Technology, Kagawa College, Japan; *J Nomoto, T Yamamoto*, Kochi University of Technology, Japan

Zinc oxide (ZnO) with a direct band gap energy of ~3.37eV at RT and a large exciton binding energy of ~60 meV is one of the promising materials for the optoelectronic devices. Chemical bath deposition (CBD) is performed at low temperatures (<100 °C), which allows us to use polymers as substrate materials. We have reported the successful growth of the ZnO nanorods (NRs) on the Au/SiO<sub>2</sub>/Si(100) substrates by CBD using different Zn precursors, i.e. ZnCl<sub>2</sub>, Zn(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O and Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (ZnNit) so far [1,2]. In this paper, the possibility of morphology-controlled CBD growth of ZnO NRs on the Au and ZnO:Ga (GZO) seed layers and effects of the difference in seed layer on their structural and optical properties will be discussed.

The substrate materials were (1) Au/SiO<sub>2</sub>/Si(100) wafers, (2) commercial Au/Ti/Si(100) wafers and (3) ion-plated GZO (200 nm)/glass films [3]. Mixed aqueous solutions of ZnNit and C<sub>6</sub>H<sub>12</sub>N<sub>4</sub> (HMT) were used as the CBD solutions. Growth time (t<sub>g</sub>) was varied in the range of 5-360 min.

SEM observations revealed that all the NRs on the GZO/glass substrate were aligned perpendicular to the substrate surface. Within t<sub>g</sub>=60 min, the average diameter and length of the NRs on the GZO/glass substrate increased rapidly with t<sub>g</sub>. Above t<sub>g</sub>=60 min, the average width and length of the NRs on the GZO/glass substrate were saturated at ~220 nm and ~1,000

nm, respectively. The average width and length of the NRs on the Au/SiO<sub>2</sub>/Si(100) substrates were found to be much larger than those on the GZO/glass substrates over the whole growth time. The relation between the growth time and the stress for the NRs on the GZO/glass substrates showed a complex behavior. At t<sub>g</sub>=5 min, the compressive stress of 0.9 GPa arose. The compressive stress decreased rapidly with t<sub>g</sub>. In the t<sub>g</sub> range of 10-15 min, the stress changed from compressive to tensile. After that, the stress was returned to the compressive. Above t<sub>g</sub>=30 min, the compressive stress was kept at ~0.6 GPa. Although the absolute values of the stresses were different, the growth time-stress curve for the NRs on the Au/SiO<sub>2</sub>/Si(100) substrates behaved similar to that for the NRs on the GZO/glass substrates. Regardless of the difference in the seed layer, the PL intensity ratio of the near-band-edge emission (380 nm) to the orange band emission (630 nm) became larger with t<sub>g</sub>, indicating the improvement of the crystalline quality.

This work was supported by JSPS KAKENHI Grant Number JP17K04989 and Yashima Environment Technology Foundation.

[1] T. Terasako *et al.*, Thin Solid Films **549** (2013) 292-298.

[2] T. Terasako *et al.*, Thin Solid Films **596** (2015) 201-208.

[3] J. Nomoto *et al.*, Thin Solid Films **601** (2016) 13-17.

10:20am **C3-8 Piezoelectric Coefficient and Morphology Investigation of the Wurtzite Ga-doped MgZnO Thin Films via RF Magnetron Sputtering**, *Ping-Han Lee, C Liu, J Huang*, National Cheng Kung University, Taiwan

The piezoelectric nanogenerators, which could convert the mechanical energy into electrical energy via piezoelectric effect, were considered to be the promising and environmentally friendly devices. The piezoelectric coefficient of the MgZnO thin films have been analyzed. In order to achieve higher piezoelectric coefficient precisely, introducing the Ga ions into the MgZnO thin films with the purpose of improving the crystallinity of the Ga-doped MgZnO. The thin films were deposited by the RF magnetron co-sputtering under 250 to achieve the thickness around 500nm, the ambient was controlled at argon 10 sccm and oxygen 20 sccm to reach MgZnO film with highest piezoelectric coefficient previously. The Ga content was varied by changing applied power from 30W to 80W of Ga<sub>2</sub>O<sub>3</sub> target, while maintaining a constant power of 100W of Mg<sub>0.3</sub>Zn<sub>0.7</sub>O target. X-ray Diffraction Analysis (XRD) confirmed that the Ga-doped MgZnO thin films showed great crystallinity and obtained strong preferential orientation along [0002] growth direction. Furthermore, the morphology and element constituent were examined by the Scanning Electron Microscope (SEM) and Energy Dispersive X-ray spectrometer (EDX). The optical property of diverse Ga content MgZnO thin films were investigated by UV-Vis spectrometer. We could obtain the superior piezoelectric coefficient with corresponding to the Ga content. Moreover, the optimal piezoelectric coefficient of MgZnO thin films, which will be utilized in piezoelectric nanogenerators, were measured by Piezoresponse Force Microscopy (PFM).

10:40am **C3-9 Growth of Al<sub>1-x</sub>Sc<sub>x</sub>N Thin Films for Pyroelectric and Piezoelectric Applications**, *Agné Žukauskaitė, Y Lu*, Fraunhofer Institute for Applied Solid State Physics IAF, Germany; *N Kurz*, IMTEK, University of Freiburg, Germany; *M Reusch, A Ding, L Kirste, V Lebedev, V Cimalla*, Fraunhofer Institute for Applied Solid State Physics IAF, Germany **INVITED** Aluminum nitride (AlN) is a well-established piezoelectric material used in telecommunication as well as sensing applications and is known for its good mechanical properties, high acoustic velocity, and high temperature stability. However, rather low piezoelectric coefficient  $d_{33} = 5-6$  pm/V, electromechanical coupling  $k_t^2 \approx 6$  % along with the low pyroelectric coefficient  $p^{eff} = 4-8$  μC/(m<sup>2</sup>K) limit the potential of AlN in energy-related applications such as energy harvesting from motion (piezoelectric effect) or temperature fluctuations (pyroelectric effect). In 2009 reactive magnetron sputtering was used to enhance AlN by alloying it with scandium nitride (ScN) to produce aluminum scandium nitride (Al<sub>1-x</sub>Sc<sub>x</sub>N) thin films, thus increasing the piezoelectric response by up to 400 % [1] and electromechanical coupling by up to 150-250 % [2] making this new material very attractive for a variety of applications. The pyroelectric properties of AlScN are largely unknown. However, our initial studies show an increase in  $p^{eff}$  by ~100 % for Al<sub>0.7</sub>Sc<sub>0.3</sub>N compared to AlN [3]. One of the main challenges for growth of high quality c-axis oriented AlScN is its metastability, as the parent binary nitrides AlN and ScN have wurtzite and cubic crystal structures, respectively, making the thin films prone to phase separation and elemental segregation if the growth conditions are not optimized. In this work reactive pulsed DC magnetron co-sputtering was used to deposit 1000 nm thick AlN(0001) and AlScN(0001) films on 100 mm diameter Si(001) substrates and then test structures for piezoelectric and

pyroelectric characterization were fabricated. The analysis of AlScN thin films was performed by using x-ray diffraction (XRD), piezoresponse force microscopy (PFM), Berlincourt method, and low-frequency temperature wave dynamic method. One of the most critical growth parameters is temperature [4], but process pressure, reactive gas composition, and, for example, target-to-substrate distance has to be taken into consideration as well [5]. With the goal of incorporating relatively high Sc amounts ( $x > 0.2$ ) into c-axis oriented wurtzite type  $Al_{1-x}Sc_xN$  while preventing the degradation of crystalline quality the investigation of different growth parameters and their influence on application-significant material properties will be discussed.

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11:20am **C3-11 A Simple Non-toxic Simultaneous Selenization/Sulfurization Process for the  $Cu(In,Ga)(S,Se)_2$  Thin Film Solar Cells**, *H Wei, Yuan-Chun Liang, Y Lin*, National Changhua University of Education, Taiwan

This paper proposes a simple non-toxic simultaneous selenization/sulfurization process to replace conventional toxic  $H_2Se/H_2S$  used and two-stage selenization/sulfurization process in  $Cu(In,Ga)(S,Se)_2$  (CIGSSe) solar cells. The proposed scheme involves sputter deposition of the absorber layer using Cu-In-Ga ternary targets, followed by the simultaneous introduction of selenium and sulfur vapor to produce a chalcopyrite CIGSSe film. Experiment results show that the total sulfur content of the absorber layer increases with an increase in the S/(S+Se) ratio. When S/(S+Se) ratio  $\geq 0.12$ , the sulfur content at the surface of the absorber layer was higher than inside, which contributed to an increase in the surface energy gap and Voc of the solar cell. However, a S/(S+Se) ratio that is too high was shown to undermine the quality of the CIGSSe crystallinity, cause phase segregation at the surface of the absorber layer, and lead to the formation of ordered vacancy compounds (OVCS) and secondary phases, which tend to decrease cell efficiency. The inclusion of a suitable proportion of sulfur in the absorber layer can inhibit excessive growth of the  $MoSe_2$  layer, and thereby enhance cell efficiency. At various S/(S+Se) ratios, small  $CuGaSe_2$  grains were observed at the bottom of the CIGSSe absorber layer. The highest cell efficiency obtained in this study was 12.1%, when S/(S+Se) ratio = 0.12. This degree of efficiency is close to that of our reference specimen, produced via two-stage selenization/sulfurization using toxic  $H_2Se/H_2S$  gases.

11:40am **C3-12 Thin Films for Transparent Thermoelectric Modules**, *F Correia, J Ribeiro, P Salvador*, University of Minho, Portugal; *A Mendes*, University of Porto, Portugal; *Carlos Tavares*, University of Minho, Portugal

A great amount of solar energy is wasted as heat in a photovoltaic (PV) cell, due to thermalisation of excited high energy electrons and absorption of low energy photons, which increases the temperature of the photovoltaic cell [1]. Given so, the cooling of PV cells has been target of considerable interest, using several cooling techniques [2-4], from which the thermoelectric (TE) devices merge. The use of TE devices as a generator to convert waste heat into electricity is much coveted and a viable alternative [5], and the approach to integrate these devices in PV cells has already been studied [6-7]. The main purpose of this work is to investigate ZnO-based thin films for thermoelectric generators (TEG), that are optically transparent throughout the whole device. These are intended to coat the top of solar cells. The strategy is to tune the ZnO thin films properties by cationic and anionic doping: with Ga/Al to improve the type n conductivity, with N to have an p type conductivity and Bi to decrease the thermal conductivity. For the latter, the hypothesis is that this will lend a bigger mass to the phonon vibration modes. Consequently a reduction in the thermal conductivity is expected. The production methodology of these films via magnetron sputtering will be presented, highlighting the most relevant process parameters.

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12:00pm **C3-13 Efficiency Enhancement in Dye Sensitized Solar Cells using Silver Ion Embedded  $TiO_2$  Photoanodes**, *Navdeep Kaur, A Mahajan*, Guru Nanak Dev University, Amritsar, India; *F Singh*, Inter University Accelerator Center, India; *S Kumar, D Singh*, Guru Nanak Dev University, Amritsar, India  
Effect of Surface Plasmon Resonance (SPR) phenomenon of silver nanoparticles (Ag nps) on photovoltaic parameters of Dye sensitized solar cells (DSSCs) have been studied. Silver nanoparticles are implanted onto  $TiO_2$  photo anodes of 3.2  $\mu m$  thickness by using 65 KeV  $Ag^{2+}$  ion beam at different fluences varying from  $10^{12}$  to  $10^{14}$  ions/cm<sup>2</sup>. The penetration depth of Ag nps was calculated to be around 17 nm through Stopping and range of ions in Matter (SRIM). Ag nps embedment have been confirmed through X-Ray Diffractometer (XRD), FESEM, UV-Vis and Raman Spectroscopy techniques. Photoanode containing Agnps embedded  $TiO_2$  loaded with N719 dye results in absorption enhancement leading to fabrication of improved DSSCs. Efficiency of DSSC containing Ag nps irradiated  $TiO_2$  photoanodes with fluence ions/cm<sup>2</sup> shows maximum increase as compared to the reference cell.

## Fundamentals and Technology of Multifunctional Materials and Devices

### Room Sunrise - Session C4

#### Energetic Materials and Microstructures for Nanomanufacturing

**Moderators:** Karsten Woll, Karlsruhe Institute of Technology (KIT), Ibrahim Emre Gunduz, Purdue University, USA

**2:10pm C4-3 High Surface Area Silicon Quantum Dots for Energetic Materials, Philip M. Guerieri, N Piekiet, S Adams, M Ervin, C Morris, U.S. Army Research Laboratory, USA**

“On-chip” porous silicon has now been researched for a number of years as an energetic material to augment traditional electronics performance, or provide thrust or actuation in small-scale applications. On-chip porous silicon is ideal for these applications thanks to its high energy density and MEMS fabrication capabilities. However, when you consider that “on-chip” porous silicon is, by its nature, attached to a piece of silicon wafer, the effective energy density when including the mass of the energetic portion and the mass of the inert substrate base, is much lower than the energy density of the energetic material alone. In an attempt to obtain similar performance as on-chip porous silicon, we have fabricated silicon powders with a <5nm (quantum dots) primary particle size for use as a fuel in energetic materials. Initial results demonstrated that flame speed in open channels can eclipse 1 km/s and is comparable to on-chip porous silicon. This study further investigates flame speed characterization of these particles with various oxidizers, and explores mixture of this material with various binders for additive manufacturing applications. FTIR, EDS, TEM, SEM, high speed imaging, bomb calorimetry have all been used to characterize the particles and/or energetic formulations.

**2:30pm C4-4 Investigating Transport Processes in Multilayer Films, David Adams, M Abere, C Sobczak, Sandia National Laboratories, USA**

Metallic thin film multilayers that undergo rapid, self-propagating formation reactions are of interest for several applications including advanced joining technology. The development and optimization of new materials for these applications requires a detailed understanding of mass transport, chemical reactions, heat release and thermal transport processes. With this presentation, we focus on the thermal properties of produced multilayers. Thermoreflectance techniques have been used to characterize the thermal conductivity of different Pt/Al, Co/Al and Ni/Al multilayers. The bilayer thickness dependence of cross-plane thermal conductivity has been determined for various multilayers. The results are examined in terms of conductivity through the reactant layers and the role of interfaces. The interfacial structure and composition of each system has been mapped by cross section transmission electron microscopy. The measured properties are compared with estimates derived from analytical modeling of self-propagating formation reactions. The model developed by Mann et al. (J. Appl. Phys. 1997) to predict how measured flame speeds vary with multilayer design is used to estimate the thermal and mass transport characteristics. This analytical model accounts for reactant layer thicknesses, compositional profiles near interfaces, flame temperatures, measured heats of reaction, measured activation energies, and adiabatic temperatures.

This work was supported by a Sandia Laboratory Directed Research and Development (LDRD) program. Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA-0003525.

**2:50pm C4-5 Analytical Modelling of Propagation Velocity in Non-stoichiometric and Impact Compressed Nanolaminates, Michael Abere, D Adams, Sandia National Laboratories, USA**

The ignition of sputter deposited bimetallic nanolaminate films results in rapid, self-propagating reactions. Analytical models of the measured propagation velocities have been typically performed using a framework developed by Mann et al. (J. Appl. Phys. 1997). This work seeks to expand upon this model to handle bimetal systems such as Al/Pt in which the lateral and transverse thermal conductivity are highly anisotropic. A thermal circuit model is thus employed that is shown to hold for both equimolar and non-stoichiometric compositions of Al/Pt. Furthermore, this work utilizes cross-sectional scanning transmission electron microscope

energy-dispersive X-ray spectroscopy data to calculate the Fourier coefficients in the Mann et al. model from the physical composition profile in the intermixed region. The same framework can also be applied to determine the magnitude of plastic deformation necessary after laser flyer impact to produce observed propagation velocities as much as a factor of two above the steady state velocity of NiV/Al within the impacted zone.

**3:10pm C4-6 On the Fly Mixing and 3D Printing of Al/CuO Thermite for Controlling Reactivity, Alexandra Golobic, M Durban, Lawrence Livermore National Laboratory, USA; E Duoss, Lawrence Livermore National Laboratory, USA, US; A Gash, K Sullivan, Lawrence Livermore National Laboratory, USA**

The ability to spatially control the behavior of reactive materials *within* a part is now a reality with advances in 3D printing. This vastly opens up the design space for rapidly deflagrating materials, such as pyrotechnics or thermites, to yield a precise property or dynamic performance. In order to achieve this goal, a mixing print head was used to mix an aluminum and a copper oxide ink on the fly. The mixing and printing parameters were first investigated for a stoichiometric mix of fuel and oxidizer to determine at what point the material can be assumed well-mixed. The equivalence ratio was then changed, and the critical mixing parameters established. The reactivity was characterized by printing a strip of material, then initiating the thermite and measuring the propagation velocity with a high-speed camera. Once the velocity reached a plateau, we considered the system well mixed. 3D printing was then used to make parts where the local stoichiometry, which corresponds to performance, is spatially varied. Collective effects of having incorporated features with differing reactivities were investigated.

**3:30pm C4-7 Tin-based Composites Combined with Reduced Graphene Oxide via a Simple Chemical Treatment as Anode Material for Rechargeable Lithium Ion Batteries, Yi-Zhu Wu, National Cheng Kung University, Taiwan; C Chang, National University of Tainan, Taiwan; S Brahma, J Huang, National Cheng Kung University, Taiwan**

We successfully synthesize the reduced graphene oxide/SnO<sub>x</sub> (RGO/SnO<sub>x</sub>) composites via a one-step chemical treatment with low cost and low toxicity at room temperature. In this procedure, we use the Sn(BF<sub>4</sub>)<sub>2</sub> as the precursor and NaBH<sub>4</sub> as the reducing agent to deposit the tin onto reduced graphene oxide and utilize the composite as anode material for lithium ion batteries. With different concentration of reducing agent, we can control different reduction degree of composites. This study shows that reductant concentration significantly affect the density and agglomeration of nanoparticles over the GO sheets. The average size of the nanoparticles in the composites is approximately 5 nm. The observed electrochemical performance of RGO/SnO<sub>x</sub> composite shows improved capacity (937.9 mAh/g for first cycle discharge) and good cycling ability (824.0mAh/g with 88% retention after 50 cycles.).

**3:50pm C4-8 Additive Manufacturing of a Composite Solid Propellant with High Solids Loadings, Monique McClain, I Gunduz, S Son, Purdue University, USA**

Solid propellant performance is strongly dependent on the manufacturing process. The traditional method of casting propellant limits the ability to locally vary geometry and reactivity throughout the grain and could lead to the creation of defects. Additive manufacturing (AM) has been effectively demonstrated as an alternative manufacturing process for complex hybrid propellant grains. However, methods such as jet printing, stereolithography, and fuse deposition modeling are limited by the materials that can be printed. Conventional printable materials are less reactive than baseline fuels, such as hydroxyl-terminated polybutadiene (HTPB), and high solids loadings have not been achieved, rendering the printing of high performing solid propellants unobtainable. In this work, an AM method developed in our lab was used to print ammonium perchlorate (AP) composite propellant strands at 85% solids loading. The viscosities of AP propellant mixtures were characterized to quantify printing parameters and the integrity of the samples were investigated with X-ray tomography scans. The printed AP propellant strands were burned at high pressure to determine the burning rate and were compared to cast samples with the same formulation. It was demonstrated that AM could be used to manufacture solid propellants at a solids loading comparable to current industry standards.

# Wednesday Afternoon, April 25, 2018

4:10pm **C4-9 Manufacturing and Characterization of Nanocomposite WC-based Powders**, *Abdulsalam Alhazza, L Al-Hajji, S El-Eskandarani, A Al-Rowayyeh*, Kuwait Institute for Scientific Research, Kuwait

In the present work, the structural, mechanical, chemical, and morphological characterizations of the synthesized powders and their consolidated buttons was investigated. The synthesized powders are mechanically-induced solid state mixing or synthesizing of nanocomposite WC-Co-metal oxide ( $\text{Al}_2\text{O}_3$ , MgO,  $\text{SiO}_2$ , and  $\text{ZrO}_2$ ) superhard material powders.

During the consolidation and manufacturing process, the nanocrystalline characteristic of the nanocomposite should be noticed and maintained to take advantage of the unique properties of the synthesized nanocomposites.

The consolidation of the nanocomposite powders will lead to the manufacturing full dense buttons with a very high hardness, fracture toughness, and wear resistance by synthesizing a nanosized powders and advent of fast sintering techniques.

4:30pm **C4-10 Ternary Reactive Ru/Al/X Multilayers - The Effect of Stacking Sequence on Ignition, Propagation and Microstructure Evolution**, *Christoph Pauly*, Saarland University, Germany; *K Woll*, Karlsruhe Institute of Technology (KIT), Germany; *I Gallino*, Saarland University, Germany; *M Stüber*, Karlsruhe Institute of Technology (KIT), Germany; *F Mücklich*, Saarland University, Germany **INVITED**

To date, self-propagating reactions in PVD multilayers have been extensively studied regarding their underlying mechanisms and applications. Adjusting the reaction behavior to meet the demands of an application requires a fundamental understanding of the mechanisms and transformations on the micro- and nanoscale. Interfacial reactions are found to play a key role in determining reaction parameters such as front propagation, heat release over time and ignition behavior. The majority of studies use binary samples consisting of elemental or alloyed layers where the bilayer thickness is the main design parameter while the material combination at the interface remains unchanged. By introducing a third kind of layers, the stacking sequence becomes an additional design parameter allowing us to define type and density of the interfaces.

In this study, we designed ternary reactive multilayers based on Ru/Al by partially substituting either Ru or Al for selected elements which allows us to retain the B2-structure of the product phase. The system Ru/Al shows a heat of formation and propagation velocity comparable to that of Ni/Al, however, its temperature of reaction and ignition are higher. We present how stacking sequence and ternary additions affect the properties of the self-propagating reaction. The role of interfacial solid state reactions on ignition is discussed and how this can be used to modify ignition temperatures. Strong effects of composition and stacking sequence on net propagation velocity are observed and discussed with the help of microstructural analysis of quenched reaction fronts.

## Fundamentals and Technology of Multifunctional Materials and Devices

### Room Sunrise - Session C2-1

#### Novel Oxide Films for Active Devices

**Moderators:** Marko Tadjer, Naval Research Laboratory, USA, Vanya Darakchieva, Linköping University, Sweden

**8:00am C2-1-1 Characteristic of the bionic synapse on Lithium Aluminate Non-Volatile Resistive Random Access Memory, Wan-Ching Su, T Chang, Y Hung, B Yan, S Huang, Y Tsao, T Tsai,** National Sun Yat-Sen University, Taiwan

This topic investigate the Characteristic of bionic synapse by non-voltage resistance random memory(RRAM). The material is aluminate lithium oxide as isolator. In operating process of RRAM the reset process reveals two reset stages. Moreover, The methods of measure it either DC voltage or Pulse have been applied to perform lithium aluminum oxide RRAM reveals the HRS resistance changing continuously. Applying the ionic diffusion and the titanium nitride attract is discussed ion model of lithium-ion, the phenomenon of the wide resistance value, and utilizing oxygen ions model. The redox reset resistance of lithium wire are explained for. Final, With Pulse voltage applied on lithium aluminum oxide RRAM can induce a bionic brain behavior: Spike-Timing-Dependent Plasticity.

**8:20am C2-1-2 Compared with the Different Thickness of Switch Layer on Resistive Random Access Memory, Chih-Cheng Yang, T Chang, W Chen, C Lin, H Zheng, Y Chien,** National Sun Yat-Sen University, Taiwan

Resistive random access memory(RRAM) is one of the next generation memory due to its low fabricated cost, structure simple, and high speed switch. In this work, the three HfO<sub>2</sub> thickness of Pt/HfO<sub>2</sub>/TiN RRAM device was used to compared the IV curves. RRAM device was deposited by sputtering. After the device fabrication, the forming voltage and IV switch cycle was measured to compared with the different thickness. Moreover, IV curves was used to study the on/off ratio and the current fitting was used to identify the current conduction mechanism. Finally, the conduction model was proposed to explain the on off ratio and current conduction mechanism.

**8:40am C2-1-3 Investigating Abnormal Hump Under Positive Bias Temperature Stress for Hydrogenated a-InGaZnO Thin Film Transistors, Yu-Chieh Chien, T Chang, T Tsai, H Chiang, Y Yang, Y Tsao, M Tai,** National Sun Yat-Sen University, Taiwan

Amorphous indium gallium zinc oxide (a-InGaZnO) is one of the most promising candidate for next generation electronics. A-InGaZnO is well known for its superior electrical characteristic, including high uniformity, high mobility (~10cm<sup>2</sup>/Vs), low leakage current (~10<sup>-20</sup>A). In addition, it can be fabricated by RF-sputtering at room temperature for application in transparent flexible displays. However, rapidly grown of display industry, including active-matrix liquid crystal display (AM-LCD) and active matrix organic light-emitted diode (AM-OLED), even 3D display technology, then enhance the requirement of carrier mobility. Thus, hydrogen were proposed to enhance device carrier mobility. However, reliability test after introducing hydrogen atoms need to be evaluated cautiously. In this investigation, a-InGaZnO TFTs after hydrogen plasma treatment (HPT) under positive bias temperature stress (PBTs) was compared to un-treated devices. An abnormal hump under PBTs condition was observed, in addition, the hump phenomenon only occurs in HPT devices. Hydrogen migrates to SiO<sub>x</sub> etching stop layer (ESL) induce positive fixed oxide charge was proposed and explained the degradation. Furthermore, different experiment conditions and COMSOL simulation were carried out to further verified the proposed model.

**9:00am C2-1-4 Optical and Electronic Properties of Monoclinic Ga<sub>2</sub>O<sub>3</sub> Unravelling, Mathias Schubert,** Linköping University, Sweden, USA; *A Mock, R Korlacki, S Knight,* University of Nebraska-Lincoln, USA; *V Darakchieva,* Linköping University, Sweden; *B Monemar,* Linköping University, Sweden; *Y Kumagai,* Tokyo University of Agriculture and Technology, Japan; *K Goto,* Tamura Corp., Japan; *M Higashiwaki,* National Institute of Information and Communications Technology, Japan

The stable monoclinic phase of Ga<sub>2</sub>O<sub>3</sub> is currently at the forefront of research for high-power electronic and high-energy photonic applications due its large break down voltage and large band gap energy. Precise knowledge of its optical and electronic properties is crucial for further progress. Due to its monoclinic crystal symmetry, traditional techniques for

measurement and calculation of solid state physics properties require careful reconsideration. In our presentation, we reveal how phonons [1], free carrier excitations [1], band-to-band transitions [2], optical constants [2], and exciton formation [2] differ fundamentally from previous front running semiconductors such as zincblende or wurtzite structure group-III group-V compounds. We demonstrate how traditional linear optical spectroscopy methods such as generalized ellipsometry and the optical Hall effect must be expanded to determine phonon, free carrier, and band-to-band transition characteristics from experiment, and we compare our findings with results from density functional theory calculations [2]. We discuss band ordering and polarization selection rules for band-to-band transitions, the peculiarities of valence and conduction band effective mass parameters, the breaking of phonon mode propagation degeneracies, the consequences for propagation of free charge carrier modes, and the need for the reformulation of the Lyddane-Sachs-Teller relationship [3]. We obtain a new description of dielectric function tensor properties from the Terahertz to the Deep Ultraviolet, and we revise previous incomplete phonon and band-to-band properties assignments [4,5]. [1] M. Schubert et al., Phys. Rev. B 93 (2016) 125209. [2] A. Mock et al., arXiv:1704.06711v1. [3] M. Schubert, Phys. Rev. Lett. 117 (2016) 215502. [4] C. Sturm et al., Phys. Rev. B 94 (2016) 035148. [5] C. Sturm et al., APL Materials 3 (2015) 106106.

**9:20am C2-1-5 Ga<sub>2</sub>O<sub>3</sub> for Ultra-High Power Rectifiers and MOSFETs, Stephen Pearton, F Ren, J Yang, P Carey,** University of Florida, USA; *M Tadjer, M Mastro,* Naval Research Laboratory, USA

**INVITED**

Gallium Oxide (Ga<sub>2</sub>O<sub>3</sub>) is emerging as a viable candidate for certain classes of power electronics, solar blind UV photodetectors, solar cells and sensors with capabilities beyond existing technologies due to its large bandgap. The performance of technologically important high voltage rectifiers and enhancement-mode Metal-Oxide Field Effect Transistors benefit from the larger critical electric field of β-Ga<sub>2</sub>O<sub>3</sub> relative to either SiC or GaN. Reverse breakdown voltages of over 1kV for β-Ga<sub>2</sub>O<sub>3</sub> have been reported by several groups, either with or without edge termination. The on-off ratios ranged from 3x10<sup>7</sup> to 2.5x10<sup>6</sup> for this range of biases and showed only a small dependence on temperature in the range 25-100°C. The metal-oxide-semiconductor field-effect transistors (MOSFETs) fabricated on Ga<sub>2</sub>O<sub>3</sub> to date have predominantly been depletion (d-mode) devices, with a few demonstrations of enhancement (e-mode) operation. The channels have been undoped, Si, Sn or Ge -doped and HfO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> have been the most widely used dielectrics. Si ion implantation has been employed to improve source/drain resistance in some cases.

**10:00am C2-1-7 Fabrication and Characterization of Pulsed-Laser Deposited Ba<sub>0.8</sub>Ca<sub>0.2</sub>Ce<sub>x</sub>Ti<sub>1-x</sub>O<sub>3</sub> (BCCT) Thin Films, Cristian Grijalva,** The University of Texas at El Paso, USA; *J Jones,* Air Force Research Laboratory, Materials and Manufacturing Directorate, USA; *R Chintalapalle,* The University of Texas at El Paso, USA

Intrinsic and doped barium titanate (BaTiO<sub>3</sub>) thin films have drawn considerable recent interest due to their second order nonlinear response, ferroelectric properties, and electro-optic properties. Co-doping approach, which proved to be quite successful with many of the multifunctional materials, has been attractive to tailor the structural, optical, electrical and mechanical properties of barium titanate ceramic thin films. Therefore, in the present work, the Ca,Ce co-doped barium titanate materials were considered to obtain tunable optical properties. Ba<sub>0.8</sub>Ca<sub>0.2</sub>Ce<sub>x</sub>Ti<sub>1-x</sub>O<sub>3</sub> (BCCT) thin films with fixed Ca content and variable Ce content were fabricated using pulsed-laser deposition (PLD). While such BCCT thin films are anticipated to have applications in electro-optic and memory devices, a detailed characterization has been performed to understand the effect of Ce on the structural and optical properties of resulting BCCT films. BCCT films with a nominal thickness of 90 nm were deposited onto quartz and low-impedance Silicon wafers heated to 250 °C. X-ray diffraction (XRD), spectroscopic ellipsometry (SE) and nano-mechanical were performed to understand the effect Ce on the structure and properties. The results indicate that the BCCT films were amorphous. All the BCCT films were optically transparent. The band gap decreases with increasing Ce content. The results and analyses will be presented and discussed in the context of utilizing these films in contemporary electronic and optical device application.

**10:20am C2-1-8 Thermo-Chemical Stability Evaluation of Titanium Doped β-Ga<sub>2</sub>O<sub>3</sub> Thin Films, S Manandhar, A Battu, Ramana Chintalapalle,** University of Texas at El Paso, USA

Gallium oxide (Ga<sub>2</sub>O<sub>3</sub>), one among the wide band gap oxides, has drawn the attention of scientific and research community for its fascinating physical,



## Thursday Morning, April 26, 2018

chemical and electronic properties, which can be readily utilized in numerous technological applications.  $\text{Ga}_2\text{O}_3$  with a band gap ( $E_g$ ) of  $\sim 5$  eV is an ideal candidate for utilization in the field of electronics, optoelectronics, spintronics, gas sensing, and ultraviolet photo detectors. Specifically,  $\beta$ - $\text{Ga}_2\text{O}_3$  is stable at very high temperatures and has shown to function as oxygen sensor at high temperatures ( $>700^\circ\text{C}$ ). We recently demonstrated improvement of response characteristics time and sensitivity towards oxygen sensing at high temperature using metal doped  $\beta$ - $\text{Ga}_2\text{O}_3$ . However, a fundamental study of thermo-chemical stability of metal doped  $\beta$ - $\text{Ga}_2\text{O}_3$  is quite important to predict the thermodynamic stability and performance of such materials in extreme environments. In this work, we performed a detailed thermal study to understand the effect of extreme environment on titanium (Ti) doped  $\beta$ - $\text{Ga}_2\text{O}_3$  (GTO). The GTO films with variable Ti content were deposited by co-sputtering. The real environment condition for sensor ( $>700^\circ\text{C}$ ) application was simulated to understand the effect of temperature on the crystal structure, electronic properties and oxidation states of Ti doped  $\beta$ - $\text{Ga}_2\text{O}_3$ .

## Fundamentals and Technology of Multifunctional Materials and Devices

### Room Sunrise - Session C2-2

#### Novel Oxide Films for Active Devices

**Moderators:** Marko Tadjer, Naval Research Laboratory, USA, Vanya Darakchieva, Linköping University, Sweden

**1:30pm C2-2-1 Investigation of Negative Bias Temperature Instability under Illumination on P-type Low Temperature Poly-crystalline Silicon Thin Film Transistors, Shin-Ping Huang, T Chang, A Chu, W Su, W Chen, National Sun Yat-Sen University, Taiwan; Y Chen, Y Shih, National Taitung University, Taiwan; Y Zheng, Y Wang, National Sun Yat-Sen University, Taiwan**

This work investigates the effect of the negative bias temperature instability (NBTI) (temperature range from room temperature to 100°C) under illumination in p-channel low temperature poly-crystalline silicon thin film transistors (LTFS TFT), employing back-faced 20000 lux white light.

Experimental results show an apparent  $V_t$  shift after NBTI with illumination during the stress time since the trapped charge in insulator layer causes the degradation.

Moreover, off current in both of the linear and saturation region shows that the degradation is affected by strong and weak field effect under illumination.

**1:50pm C2-2-2 Mechanism of Reset Process with Varying Compliance Current in High-k Spacer Resistance Random Access Memory, Yi-Ting Tseng, T Chang, W Huang, Y Guo, T Chang, W Chen, National Sun Yat-Sen University, Taiwan**

In this study, a problem of forming voltage increased during device cell scale-down in resistance random access memory (RRAM) has been solved by adding high dielectric constant (high-k) material as a side-wall (spacer) structure. In contrast, a normal side wall material is used low dielectric constant material. Electric characteristic of high-k spacer RRAM shows a great electric behavior and is the same with a normal RRAM. High resistance state (HRS) of reset process of values obviously increased during varying compliance current of set process from 1mA to 10mA in high-k spacer RRAM. However, HRS didn't clearly different change during controlled compliance current in normal RRAM. Varying compliance current of set process is as applying different energy to switch resistance. Then, AC pulses was applied to switch resistance for verifying that relationship. AC pulse of rising time was controlled between 10 $\mu$ s to 90 $\mu$ s for applied reset process. As a result, value of HRS increases with increasing rising time. Mechanism of high-k spacer RRAM is dominated by Schottky emission. From intercept and slope of Schottky emission, HRS can be analyzed further for insulator of barrier and dielectric constant.

**2:10pm C2-2-3 Improve Reliability of Complementary Resistive Switching Induced by Carbon Dopant in Indium-Tin-Oxide as The Insulator in Resistive Random Access Memory, Chun-Chu Lin, T Chang, W Chen, Y Tseng, S Huang, H Zheng, National Sun Yat-Sen University, Taiwan**

Among these possible candidates, resistance random access memory (RRAM) is recognized as the most capable of replacing flash memory due to its non-volatility, simple structure, and easy integration into CMOS fabrication. Previous experiments investigate the Complementary resistive switching (CRS) characteristic by co-sputtering indium-tin-oxide (ITO) with oxygen ( $O_2$ ) gas as the insulator. However, Pt/ITO( $O_2$ )/TiN RRAM device shows poor endurance of CRS I-V characteristic. In this work, double insulator layers are used by co-sputtering ITO with  $O_2$  and carbon to improve endurance of CRS characteristic as the Self-Rectifying Cell (SRC) and also resistance switching (RS) properties of RRAM. The chemical bonds of this Pt/ITO( $O_2$ )/ITO(C)/TiN device was also investigated with FTIR spectrum measurement. Moreover, endurance test was also carried out to confirm its RS stability and fast I-V measurement was applied to make sure its CRS I-V curve when giving the pulse. Finally, a conduction model was proposed to clarify the RS characteristics, and support the Pt/ITO( $O_2$ )/ITO(C)/TiN device as appropriate for Self-Rectifying Cell (SRC).

**2:30pm C2-2-4 Study on the Characteristic of Cobalt Silicide Electrode Resistive Random Access Memory, Wen-Chung Chen, T Chang, T Tsai, Y Zhang, S Huang, Y Lin, C Lin, H Zheng, National Sun Yat-Sen University, Taiwan**

Resistance random access memory (RRAM) is one of the promising next-generation nonvolatile memory devices due to its simple metal insulator-metal structure and its ability for high density integration. Furthermore, RRAM also has superior characteristics such as low operation voltage, fast operation speed, and nondestructive reading, and has attracted much interest by many academics and industries. On the other hand, cobalt silicide is popular to use on the semiconductor industry. It can use to reduce the contact resistance. In addition, the Self-alignment process can replace the lithography process by cobalt silicide. In this work, cobalt silicide was deposited as the top electrode, silicon oxide doped hafnium deposited as the translation layer, and the TiN was deposited as the bottom electrode. Though the device shows poor endurance, it has a large memory window by over reset. After reducing the thickness of cobalt silicide, the device shows a great improvement as compared to the device of thick cobalt silicide. Finally, we propose a model to explain the characteristic of cobalt silicide electrode on the resistive switching behaviors.

**2:50pm C2-2-5 Material and Device Engineering for Gallium Oxide Electronics, Siddharth Rajan, The Ohio State University, USA INVITED**

The presentation will give an overview of our recent accomplishments and the future outlook for high-performance  $\beta$ -Ga $_2$ O $_3$  based semiconductor materials and devices. The ultra-wide band gap semiconductor  $\beta$ -Ga $_2$ O $_3$ , is attractive for applications in next-generation high frequency and power switching devices due to availability of large area substrates, large breakdown field, and good electron transport properties. We will first discuss the main research opportunities and potential applications for these devices. This will be followed by an overview of our experimental results on molecular beam epitaxial (MBE) growth of  $\beta$ -Ga $_2$ O $_3$ ,  $\beta$ -(Al,Ga) $_2$ O $_3$ , and Si doping. We will then discuss our work on growth and characterization of heterostructures based on  $\beta$ -(Al,Ga) $_2$ O $_3$ /  $\beta$ -Ga $_2$ O $_3$  and the demonstration of modulation-doping in  $\beta$ -(Al,Ga) $_2$ O $_3$ /Ga $_2$ O $_3$  channels. We will then discuss recent our experimental device results on delta-doped and modulation-doped field effect transistors with high current density and transconductance, and discuss their DR, pulsed, and RF performance. We are grateful to Department of the Defense, Defense Threat Reduction Agency (Grant HDTRA11710034), ONR EXEDE MURI program, and the OSU Institute for Materials Research Seed Program for funding.

[1] Krishnamoorthy, S., Xia, Z., Bajaj, S., Brenner, M., & Rajan, S. (2017). Delta-doped  $\beta$ -gallium oxide field-effect transistor. *Applied Physics Express*, 10(5), 051102.

[2] Krishnamoorthy, S., Xia, Z., Joishi, C., Zhang, Y., McGlone, J., Johnson, J., ... & Rajan, S. (2017). Modulation-doped  $\beta$ -(AlO. 2Ga0. 8) 2O3/Ga2O3 field-effect transistor. *Applied Physics Letters*, 111(2), 023502.

**3:30pm C2-2-7 The Ultra-violet Light Effect on the Off-state Current of InGaZnO Thin Film Transistor with the Different Structure, Yu-Ching Tsao, T Chang, Y Tsai, W Su, S Huang, Y Chien, National Sun Yat-Sen University, Taiwan**

In this work, we discuss the ultra-violet (UV) light effect in amorphous InGaZnO $_4$  (IGZO) thin film transistor with different drain metal capping area and different active layer thickness. An asymmetric off-state current of transistors in forward and reverse sweep due to different ultra-violet light exposure region and length. An obvious off-state current can be found as a result of a source barrier lowering causing by UV light exposure near the source side. Different off-state current can also be found in different thickness of IGZO active layers. A model is also introduced to interpret this phenomenon.

**3:50pm C2-2-8 Study on the Characteristics of Device in Copper Ion Movement during Operation Process in Conductive-Bridging Random Access Memory, Ming-Hui Wang, T Chang, Y Tseng, H Zheng, C Wu, S Huang, National Sun Yat-Sen University, Taiwan**

In this experiment, Materials commonly used in semiconductor processes hafnium oxide as the insulating layer, The top electrode is copper, bottom electrode use titanium nitride, Conductive-Bridging Random Access Memory is metal-insulator-metal structure. The device operational process happen abnormal phenomenon, In the resistance decrease process (SET) will first decrease after increase and then decrease until Low Resistance State similar to the negative differential resistance, and this feature is closely related to CBRAM conduction mechanism, because copper ion

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diffusion to the insulation lead to dielectric constant change, and then condition similar negative differential resistance phenomenon.

4:10pm **C2-2-9 The Degradation Mechanism of Tungsten Electrode on HfO<sub>2</sub>-based Resistance Random Access Memory (RRAM)**, *Hao-Xuan Zheng, T Chang, T Chu, M Wang, C Lin, C Yang*, National Sun Yat-Sen University, Taiwan

In this study, using tungsten as the role of the electrode in Resistive Random Access Memory (RRAM) has good characteristics. In addition, on/off ratio achieves two orders, and the 85°C Retention test also has a very good stability. However, after a number of the pulse cycle, there has a significant degradation in this kind of device, which is an uncommon phenomenon from the RRAM which electrode is made by inert elements such as platinum. RRAM can cause significant effects on endurance and retention due to the difference in the electrode material. By clarifying the switching mechanism and conduction current fitting, we can find that the on state conduction mechanism is transformed from Poole-Frenkel emission to Schottky emission. In addition, we use the Energy Dispersive X-Ray Spectroscopy (EDS) analysis and proposed a physical model to explain the main cause of degradation, due to oxygen ions diffusing to the electrode.

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## Fundamentals and Technology of Multifunctional Materials and Devices

Room Grand Hall - Session CP

### Symposium C Poster Session

#### CP-2 Effect of Nitrogen Content on Structure and Properties of MoN<sub>x</sub> Coatings, *Jian Wang*, University of New South Wales, Australia

Molybdenum nitride (MoN<sub>x</sub>) coatings were deposited onto AISI M2 tool steel substrates (hardened to HRC 60) by closed field unbalanced magnetron sputtering ion plating (CFUMSIP) and controlled by means of a closed-loop optical emission monitor (OEM), which was used to control the nitrogen content. The structure of the coatings was investigated by X-ray photoelectron spectrometry (XPS), X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The analysis showed that increased nitrogen content led to a transformation from bcc Mo phase to fcc Mo<sub>2</sub>N phase and then to fcc MoN phase as the OEM level was decreased. The mechanical and tribological properties were evaluated by nano-indentation, wear and scratch testing. During the wear test, the Mo<sub>2</sub>N exhibited the lowest coefficient of friction. Further, during the scratch test, the Mo coating failed at the lowest load. This presentation will relate the processing conditions to the structure and hence mechanical behavior of these coatings.

#### CP-3 Stress Metrology for G6 and Larger Flat Panel Displays, *Wojtek Walecki*, Frontier Semiconductor, USA; *W Hung*, Frontier Semiconductor, USA, United States of America; *D Kim*, Sejong University, Korea

We report novel photo-elastic method for measurement of the stress in polyimide (PI) based flat panel displays (FPD) and flexible displays (FD) structures. Method is based on measurement of the change of the state of polarization of the light undergoing reflection (or transmission) in the structures containing PI layer(s). Commonly used FPD and FD contain layers of 5 μm or thicker PI layers. PI has stress optic coefficient of about 3.4E-10 Pa<sup>-1</sup> [1], which is almost 100 times larger than glass [2]. This allows easily measurement of stress with sensitivity of the order of 5 MPa and less. We present apparatus for local stress measurements having lateral resolution of 3 cm, and stress resolution of 5 MPa. Presented tool gives promise to become capable of measuring glass panels of the size 1.5 m x 1.85 m does not contained any moving parts. Our tool can be combined with more traditional stress induced deflection based stress measurement [3]. We discuss also methods of numerical analysis of optical data, including stress separation algorithm optimized for this problem, and practical problems related to analysis of the data.

[1] Noe, Susan Cunningham. *A prism coupling study of optical anisotropy in polyimide including moisture, stress, and thickness effects*. Diss. Massachusetts Institute of Technology, 1992.

[2] Sun, Lan, and Samad Edlou. "Low birefringence lens design for polarization sensitive optical systems." *Proc. SPIE*. Vol. 6289. 2006.

[3] Walecki, Wojtek *J Stress Metrology for Flat Panel Displays G6 and Bigger*. Abstract submitted to Photonics West 2018 SPIE OPTO (PW180).

#### CP-4 Hydrogen Barrier Properties of Diamond-like Carbon Coatings, *Motonori Tamura*, University of Electro-Communications, Japan

The hydrogen barrier properties of the coatings of diamond-like carbon (DLC) were evaluated. Using plasma chemical vapor deposition and sputtering, DLC coatings were deposited on Type 316L stainless steels. The hydrogen permeation rate was reduced to 1/1000 or lower by the DLC coatings. The DLC coatings with high hydrogen content had high hydrogen barrier function. For hydrogen diffusion in coatings, the movement of atoms through hydrogen trap sites such as pores in coatings, and crystal defects such as dislocations, is important. The DLC coatings are amorphous, and there are both sp<sup>3</sup> and sp<sup>2</sup> bonds, and excess hydrogen could be found in the interstitial space and the hydrogen trap sites. In the DLC coatings with high hydrogen content, these hydrogen trap sites are likely already filled with hydrogen atoms, and the movement of new hydrogen atoms could be limited.

**CP-5 Effect of N<sub>2</sub> Flow Rate on the Properties of TiN film on Si Substrate for Thermal Detector Application, *Yi-Ching Huang*, *K Lin*, *Y Lai*, National Nano Device Laboratories, National Applied Research Laboratories, Taiwan**  
Titanium nitride (TiN) is a suitable material for the mirror film of thermal detector due to its high IR reflectivity property. TiN thin films have been deposited on p-type Si (100) substrate with different nitrogen flow rate by

magnetron sputtering system. Increasing the N<sub>2</sub> flow rate played a significant factor in controlling the properties of TiN films. In the study, the surface performance, phase, element ratio, and the optical and electrical properties of the TiN films will be characterized by scanning electron microscopy (SEM), x-ray diffraction (XRD), x-ray photoelectron spectrometer (XPS), Fourier Transform Infrared Spectroscopy (FTIR), and current-voltage (I-V) measurement. The results indicated that the TiN film with 40 sccm N<sub>2</sub> flow rate had high reflection (88%) in the IR range and exhibited the lower sheet resistance and high I<sub>on</sub>/I<sub>off</sub> ratio.

#### CP-6 Graded Multilayer Thin Film of BaTiO<sub>3</sub>/PVDF with High Energy Storage Density, *XiaoHui Wang*, Tsinghua University, China

Organic-inorganic 0-3 nanocomposites, which combine the potentially high dielectric strength of the organic matrix and the high dielectric permittivity of the inorganic filler, are extensively studied as energy-storage dielectrics in high-performance capacitors. To obtain high dielectric constants, a large volume fraction of the inorganic component is necessary, but this will frequently deteriorate the dielectric (breakdown) strength and thus limit the energy density value of the overall nanocomposite. In this study, a graded multilayer BaTiO<sub>3</sub>/poly(vinylidene fluoride) thin film structure is presented as a means to achieve both a higher breakdown strength and a superior energy-storage capability. Key to the process is the sequential deposition of uniform dispersions of the single component source, which generate a blended PVDF-BTO-PVDF structure prior to full evaporation of solvent, and thermal treatment of the dielectric. The result is a 2-2 like sandwich structure with partial 0-3 character, seamless interfaces between layers and a concentration gradient of the BTO. The central layer designed to provide the high electric displacement, is composed of high volume fraction 6-10 nm BTO nanocrystals produced by a TEG-sol method. The outer layers of the structure are predominantly PVDF, with a significantly lower volume fraction of BTO, taking advantage of the higher dielectric strength for pure PVDF at the electrode-nanocomposite interface. The film is mechanically flexible, and can be removed from the substrate, with total thicknesses in the range 1.2 – 1.5 μm. Parallel plate capacitance devices exhibit highly improved dielectric performances, compared to reported values for BTO-PVDF 0-3 nanocomposites, with low-frequency permittivity values of 20-25, a maximal discharged energy density of 19.4 J/cm<sup>3</sup> and dielectric breakdown strengths of up to 495 kV/mm.

#### CP-7 Synthesis of Bi<sub>2</sub>O<sub>3</sub>:TiO<sub>2</sub> Nano Structured Thin Films for Photocatalytic Applications, *M Calheiros*, *F Correia*, *J Marques*, *Carlos Tavares*, University of Minho, Portugal

The increasing scarcity of potable water has served as motivation for the development of decontamination processes. Photocatalytic degradation is one of the most viable processes compared with conventional ones. This process uses the UV radiation effect to produce hydroxyl radicals, with the assistance of a photocatalyst. The most commonly used catalyst is TiO<sub>2</sub> semiconductor, characterized by its low toxicity and high chemical stability. This work aims to synthesize Bi<sub>2</sub>O<sub>3</sub> thin films with fibrous morphology for subsequent functionalization with a top TiO<sub>2</sub> thin film. A Hastelloy B3 thin film was used as an interface layer between the glass substrate and the Bi seed layer in order to promote some interfacial roughness and improve film adhesion. The growth of Bi<sub>2</sub>O<sub>3</sub> thin films was performed by magnetron sputtering and adapted to abide the vapor-liquid-solid (VLS) mechanism, mainly concerning its 3D growth morphology and its high roughness templates. Subsequently, the TiO<sub>2</sub> photocatalytic thin films were deposited onto the Bi<sub>2</sub>O<sub>3</sub> thin films. SEM observations revealed a pine-tree morphology for the Bi<sub>2</sub>O<sub>3</sub> nano structures, with an enhanced surface area. The photocatalytic efficiency assessment was performed by conducting an assay using methylene blue dye as the pollutant and a solar radiation simulator. The tests show that the thin films of Bi<sub>2</sub>O<sub>3</sub>:TiO<sub>2</sub> are more efficient at degrading the pollutant when compared with the TiO<sub>2</sub> thin films.

#### CP-8 Improvement of Mechanical Properties in 3D Printed Ceramic Core, *Hye-Yeong Park*, *B Kim*, *G Cho*, *E Kim*, *Y Jung*, Changwon National University, Republic of Korea; *J Zhang*, Indiana University Purdue University Indianapolis, USA

Ceramic core employed in a precision casting process is typically not recyclable, which reduces productivity and increases production cost. Therefore, a new fabrication process for ceramic core combined with 3D printing process and organic-inorganic binder conversion process was proposed in our previous study. However, the core made of coarse mullite bead (average particle size: 250 μm) did not develop sufficient green and firing strengths due to the porosity and pore size. Therefore, to improve the mechanical properties, especially the strength, the packing density of

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ceramic core was increased, through mixing fine mullite powder (average particle size: 16 $\mu$ m) and zircon flour (average particle size: 43 $\mu$ m) with coarse mullite bead. Green bodies with the two types of poly vinyl alcohol (PVA), which have the same molecular structure with a large difference in their boiling points were 3D printed. Then the samples were heat-treated at 250°C to evaporate the PVA with a lower boiling point. The heat-treated core samples were dipped into the inorganic precursor, and dried and heat-treated at 1000°C for the organic-inorganic conversion process. Through the combination of starting powders, the compact density of the sample was increased and the pore size was reduced, resulting in an increase in the inorganic binder coating efficiency and an improvement in the glassification conversion efficiency. The study demonstrates the feasibility of fabrication of ceramic core with excellent strength through 3D printing process.

**CP-9 Enhanced Efficiency of Perovskite Solar Cells with Ferroelectricity,** *T Nguyen, S Shin, S Kim, H Choi, ChungWung Bark*, Gachon University, Republic of Korea

Perovskite solar cells (PSCs), which emerged as tremendously attractive devices in thin-film photovoltaic technology to utilize renewable energy sources, have been improved with the unprecedented breakthrough in recent years. Ferroelectric materials with a vast array of intriguing electrical properties have been applied in photo-related devices; however, there was rarely that these materials appeared in solar cell device configuration. In this work, mesostructured TiO<sub>2</sub> combined lanthanum bismuth titanate (BLT) nanoparticles in a combination with perovskite CH<sub>3</sub>NH<sub>3</sub>PbI<sub>3</sub> light absorber is capable of maximizing the absorbed visible light. Interestingly, the robust spontaneous electrical polarization of these ferroelectrics under applying positive bias voltage promotes the desirable separation of photoexcited carriers and drives the charge transportation that contributes to high-efficiency PSCs. Application of uniform nano-sized BLT powders through high-energy ball milling process and perovskite layer fabricated by two-step solution deposition technique will pave the way for fabricating hybrid organic-inorganic perovskite solar cell with high solar energy conversion in the coming years.

**CP-10 Improvement in Hygroscopicity of Inorganic Binder through Dual Coating Process,** *Hyun-Hee Choi, H Lee, G Cho, E Kim, Y Jung*, Changwon National University, Republic of Korea; *J Zhang*, Indiana University-Purdue University Indianapolis, USA

In a conventional sand casting process, the mold is manufactured by mixing ceramic materials and organic binders, which is widely used in foundry industry due to the simple manufacturing process and low production cost. However, it is difficult to form complicated products since the organic binders are decomposed and the defects in the mold are generated during casting at high temperatures. In order to solve these problems, organic-inorganic binder conversion process has been proposed. One issue in the process is that, when stored at room temperature for a long time before heat treatment, the mold strength is reduced and/or the mold is fractured, which is caused by the hygroscopicity of the water-soluble inorganic binder. Therefore, in this study, a dual coating process was proposed and applied to reduce the hygroscopicity of the inorganic binder in preparing the casting mold. The prepared sample was dipped into a solution of inorganic binder precursor (TEOS: SiO<sub>2</sub> precursor and NaOMe: Na<sub>2</sub>O precursor), and then dipped into a solution of water-insoluble organic binder after a drying process. Finally the sample was heat-treated at 1000 °C to generate a glass phase by organic-inorganic conversion process. The contact angle of the sample with the water-insoluble organic binder was increased, while it was impossible to measure the contact angle in the conventional sample. It was confirmed that the green and firing strengths were maintained by the water-insoluble organic binder coating layer. The effects of the holding time and humidity at room temperature on the strength and microstructure of the mold were investigated, in terms of organic binder species employed in the dual coating process.

**CP-11 Synthetic Parameter Influence on Morphological and Electrochemical Properties of Porous NiO Thin Films Prepared by Chemical Bath Deposition,** *Jung-Hoon Yu, H Yang, R Jeong, J Lee, D Kim, K Hwang, H Seo, S Nam, J Boo*, Sungkyunkwan University, Republic of Korea  
Chemical bath deposition (CBD) is an advantageous thin film deposition technique for depositing compound semiconductors at low temperature. In this paper, nickel oxide (NiO) thin films were prepared by CBD method under aqueous solution containing nickel sulfate, potassium persulfate, and ammonia water at room temperature. Prepared NiO thin film has porous structure with two dimensionally networked nanoflake arrays. In this process, concentration of ammonia water and nickel sulfate significantly affect on morphological features. Increase of ammonia

concentration cause the growth kinetics until 30 ml of ammonia. Over 30 ml, however, it tended to not only decrease the thickness of films but also change the growth direction of flakes. In addition, lower concentration of nickel sulfate form a thicker NiO thin film but high concentration forms a lower thickness. This phenomenon is derived from Oswald ripening between crystal growth and aggregation at the CBD process. Each prepared NiO thin films were characterized by electrochromic measurement to apply on electrochromic window. Optimized NiO thin film shows good electrochromic performance with fast switching speed (11.0 s and 7.5 s), high optical modulation ( $T_{bleaching}$ : 80% and  $T_{coloring}$ : 17 %) and high cycling durability (over 5000 cycle).

**CP-12 Characteristics of Perovskite Solar Cells Fabricated by using Lead Free Perovskite,** *S Shin, C Bark, HyungWook Choi*, Gachon University, Republic of Korea

Lead halide perovskite is an excellent candidate for use as a light harvester in solar cells. The perovskite structure (CH<sub>3</sub>NH<sub>3</sub>)PbX<sub>3</sub> (X = halogen) consists of organic components at cuboctahedral sites and inorganic components at octahedral sites, and perovskites exhibit the chemical properties of the organic component. Solid-state hybrid organic-inorganic solar cells often employ a layered structure of nanoparticulate titania, an organometal halide perovskite, and a spiro-MeOTAD hole transport material (HTM). One concern, however, is the potential toxicity of lead, an important component of conventional perovskite solar cells. Currently, the most likely substitute is a tin, and it is a Group 14 metal, similar to lead. In this paper, we develop a new type of perovskite photoresist for non-toxic perovskite solar cell applications. This is also a non-toxic material for solar cells compared to conventional materials used for perovskite solar cells. The perovskite precursor solution was prepared by dissolving CH<sub>3</sub>NH<sub>3</sub>I and SnI<sub>2</sub> in N-dimethylformamide (DMF). The application of the perovskite layer produced by the sequential deposition technique through the perovskite produced by using the tin is likely to replace the Pb-based perovskite, which is a safety and commercialization of the perovskite solar cell Improve technological progress.

**CP-14 The Influence of Disordered Grain Boundaries on Carrier Transport in Degenerated Polycrystalline AZO Thin Films Deposited by Magnetron Sputtering,** *Hiroki Tokunaga, T Miyata, T Minami*, Kanazawa Institute of Technology, Japan

The influence of disordered grain boundary on carrier transport is investigated for degenerated polycrystalline Al-doped ZnO (AZO) thin films prepared using two magnetron sputtering deposition (MSD) apparatuses. The AZO thin films with an Al content of 3 at.% and a thickness of 500 nm were prepared on glass substrates at a substrate temperature of room temperature (RT) in a pure Ar gas atmosphere at a pressure of 0.6 Pa using a dc and an r.f. (13.56 MHz) power supply applied either separately or in combination; dc-MSD and rf-MSD or rf+dc-MSD. When the electrical properties were evaluated by the van der Pauw method, the obtained mobility ( $\mu^{Hall}$ ) and carrier concentration ( $n^{Hall}$ ) in polycrystalline AZO thin films prepared by MSD methods exhibited the location dependences (distributions) on the substrate surface, when moved from the location corresponding to the center on the target surface to one corresponding to the erosion area on the target. The location dependences of  $\mu^{Hall}$  and  $n^{Hall}$  were also controlled by rf+dc-MSDs carried out with varying superimposed r.f. power. However, we found that the  $\mu^{Hall}$ - $n^{Hall}$  relationship resulting from those location dependences always exhibited a positive slope. In addition, we found that the main scattering mechanism, which limits the mobility of AZO thin films is attributed to grain boundary scattering caused by the reflection of electrons from the potential barrier at the grain boundary between crystallites. The obtained  $\mu^{Hall}$ - $n^{Hall}$  relationships with a positive slope in degenerated AZO thin films prepared by various MSDs always exhibited fair agreement with those calculated using Mayadas and Shatzkes (MS) theory. However, the significance and reliability of reflectivity used as a fitting parameter in the semi-classical MS theory seem questionable. Munoz's group recently reported that the increase in the resistivity of a metallic specimen must be estimated under the effect of electron scattering from disordered grain boundaries based upon Kubo formalism. Disordered grain boundaries were represented by a one-dimensional periodic array of Dirac delta functions separated by a distance producing a Krönig-Penney (KP) potential. They used Green's function built from the wave functions, which are solutions of the KP potential. In quantum theory, the positively sloped  $\mu^{Hall}$ - $n^{Hall}$  relationship in degenerated semiconductors such as AZO thin films is attributed to Anderson localization, induced by electron grain boundary scattering from disordered successive grains.

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1) C. Munoz et al., Appl. Surf. Sci. **329** (2015) 184. & Appl. Phys. Rev. **4** (2017) 011102.

**CP-15 Physical and Electrochromic Behavior of the ZnWO<sub>4</sub> Active Layer synthesized by Co-sputtering Technique for the Energy Harvesting Devices**, G Malik, S Mourya, J Jaiswal, Ramesh Chandra, Indian Institute of Technology Roorkee, India

This article presents a detailed investigation on a zinc tungstate (ZnWO<sub>4</sub>) active layer, prepared by reactive DC magnetron co-sputtering using standard Zn and W targets, on a transparent conducting glass ITO (indium doped tin oxide) substrate at various temperatures. The ITO substrate is used as a working electrode and ZnWO<sub>4</sub> active layer serve the purpose of a mixed conductor, which is a source of ions and electrons for the electrochromic devices. The physical properties of the fabricated active layer are investigated by x-ray diffraction (XRD), Scanning electron microscopy (SEM), Atomic force microscopy (AFM), and UV-Vis-NIR spectrophotometry. The quality and the elemental distribution of the active layer are confirmed using X-ray photoelectron spectroscopy (XPS). To determine the optical behavior, Variable angle spectroscopic ellipsometry (VASE) is deployed to calculate the optical constants ( $n$  and  $k$ ). Finally, the electrochemical performance of the active layer was analyzed by cycle voltammetry technique (CV). XRD diffractogram professed that the films are polycrystalline in nature with a monoclinic structure having P2/c space group. SEM micrographs and the cyclic voltammogram of the active layer revealed the surface modification and temperature dependent electrochromic response. This work is an attempt towards the "green nanotechnology" with energy harvesting for human comfort and financial benefits.

**CP-16 The Influence on Electrical Characteristics of Amorphous Indium Tungsten Oxide Thin Film Transistors with Multi-Stacked Active Layer Structure**, Kai-Jhih Gan, P Liu, D Ruan, Y Chiu, M Yu, T Chien, Y Chen, P Kuo, S Sze, National Chiao Tung University, Taiwan

A novel amorphous indium-tungsten-oxide thin film transistor with a multi-stacked active layer is well discussed in this work. A multi-layer channel is proposed to effectively enhance the carrier mobility and device stability, simultaneously. A top capping oxygen-rich a-IWO thin film is used for suppressing the plasma damage to channel layer during backchannel passivation layer deposition process or the oxygen desorption from channel layer by the backchannel passivation material. In addition, a bottom buffer oxygen-rich a-IWO thin film is deposited to avoid the oxygen vacancy formation during the following thermal process. On the other hand, a 1-nm thick WO<sub>3</sub> layer is inserted between the high-k gate insulator and the multi-stacked active layer, which plays important roles as interfacial layer for improving the interface quality and reducing the surface roughness. Besides, a HfO<sub>2</sub> dielectrics film is chosen as gate insulator for realizing low-voltage operation. In this research, the sample with multi-stacked active layer exhibits a high On/Off current ratio of  $\sim 1 \times 10^7$  for low gate leakage current, attributing to the bottom oxygen-rich thin film. Then, a high field-effect mobility of  $\sim 21 \text{ cm}^2/\text{V}\cdot\text{s}$  is achieved by a low surface roughness. Due to the good interface quality, the subthreshold swing is about 0.1 V/decade. This multi-stacked active layer structure exhibits its potential application for the future high-resolution and large-size display manufacture.

**CP-17 Assessment of Structural and Magnetic Properties of Cobalt-Iron-Nickel Thick Films on Copper Formed by Electroforming**, Scooter D. Johnson, C Joye, H Newman, N Nepal, A Kozen, S Shin, Naval Research Laboratory, USA

Co-Fe-Ni alloys form a family of soft magnetic material with a large magnetic saturation ( $\sim 20 \text{ kG}$ ) and very low coercive field ( $< 10 \text{ Oe}$ ) that can play an important role in developing micro electromechanical systems and other novel device structures. For example, integration of magnetic structures into vacuum electronic traveling wave amplifier circuits may dramatically reduce the bulk magnetic material needed in these devices, thereby producing favorable scaling opportunities in larger systems. Vacuum electronic devices have been made using ultraviolet photolithography and electroforming (UV-LIGA) to form bulk copper structures. Magnetic components to aid in directing the electron beam passing through the electromagnetic circuit are traditionally machined, brazed together and drilled out to accept the copper circuit structure. To explore the integration of magnetic materials into these circuits by additive manufacturing, we explore tuning the elemental and structural composition of the electroformed Co-Ni-Fe magnetic films. The resulting films were assessed using x-ray photoemission, x-ray diffraction, scanning

electron microscopy, vibrating sample magnetometry, and ferromagnetic resonance techniques.

Samples were formed using a sulfate electrolyte bath consisting primarily of cobalt sulfate, nickel sulfate and iron sulfate with several additives to act as a buffer, improve adhesion, and reduce stress. The pH was varied from 2.8 to 3.2, and the galvanic methods were varied. The plating was carried out in a nitrogen glovebox to exclude oxygen in the atmosphere. The effects of hydrogen annealing are also studied, since vacuum electronic devices are often hydrogen brazed at temperatures up to 1050 deg C.

Film thickness was estimated to be about 100  $\mu\text{m}$ . VSM results on initial samples suggest that the films have a good magnetic saturation value of about 19 kG with a small coercive field of about 50 Oe. The anisotropy field was estimated using a Stoner-Wohlfarth fit and suggests a high degree of magnetic anisotropy perpendicular to the film surface. XPS results indicate that the sample compositions vary in Ni and Fe content. Additional Co and Ni increases the magnetic saturation value while the coercive field remains unchanged.

**CP-18 Sputter-deposited Nanostructured Metal-Oxide Films for Hydrogen Gas Sensing**, S Haviar, Jiří Čapek, N Kumal, Š Batková, M Fialová, R Čerstvý, University of West Bohemia, Czech Republic; T Duchoň, F Dvořák, Charles University, Czech Republic

We present the study of nanostructured metal-oxide films prepared using a gas aggregation cluster source. The main advantage of the use of the cluster source is the possibility to prepare films with a high reactive area without the need for the use of wet techniques (often used for preparing nanostructured sensors). The films with the desired stoichiometry can be prepared directly without the need for subsequent thermal and/or chemical treatment.

Mixtures of tungsten oxide (WO<sub>3</sub>) and cupric oxide (CuO) were deposited by cluster source and/or by conventional reactive dc sputter deposition. Sputtering conditions were tuned to vary the chemical composition and structure of the prepared films. The prepared films were characterized by means of X-ray diffraction, scanning electron microscopy, atomic force microscopy and Raman spectroscopy. The elemental composition was determined by energy and wave dispersive spectroscopy. The oxidation state of metals was studied by X-Ray Photoemission Spectrometry. Subsequently, the films were examined for their sensorial response when assembled into a hydrogen gas sensor. Noble-metal catalysts (Pd, Pt) deposited by dc magnetron sputtering were used to support the response and to lower the working temperature.

The layers were tested for response to a time-varied hydrogen concentration in synthetic air at various temperatures. The response sensitivity and the response time were evaluated. It is shown that optimization of the structure and composition results in enhanced sensorial properties.

**CP-19 A Library of Broadband Reference Dielectric Functions, Valence Band Spectra and Raman Spectra of Epitaxial Conductive Nitride Films Grown on MgO**, S Kassavetis, T Zorba, J Arvanitidis, D Christofilos, Aristotle University of Thessaloniki, Greece; G Abadias, Université de Poitiers, France; D Gall, Rensselaer Polytechnic Institute, USA; Panos Patsalas, Aristotle University of Thessaloniki, Greece

Conductive nitrides, such as TiN, ZrN, and TaN have emerged as significant alternative materials for photonics and plasmonics, due to the combination of their electronic conductivity with their thermal and mechanical stability and refractory character. One of the major drawbacks for the implementation of these materials in plasmonics and photonics is their excessive electronic losses, which are usually originating from the small grain size of the grown films due to their high melting point. Therefore, their refractory character is both a blessing (in terms of stability) and a curse (in terms of grain size). This drawback might be overcome by growing nanostructures of the finest crystalline quality that can be achieved by epitaxial growth. Epitaxial or pseudo-epitaxial growth of most conductive transition metal nitrides can be achieved on MgO along all the principal orientations, or on c-cut sapphire, respectively. Therefore, for the design of photonic and plasmonic devices, it is of utmost importance to know the optical response and the electron density of states of epitaxial transition metal nitrides on MgO. In this work, we provide a library of: i) the dielectric function spectra of epitaxial transition metal nitrides (TiN, ZrN, NbN, TaN, MoN, WN) in the broad range 0.193-125  $\mu\text{m}$  (6.5-0.00992 eV), which were extracted by combining spectroscopic ellipsometry and FTIR reflectance/transmittance measurements, ii) the Raman spectra and the stability of these films upon exposure to intense laser beams, and iii) the experimental electron density of states of the valence band based on X-ray

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photoelectron spectra. In particular, the dielectric function spectra and Raman spectra of this library may also be used as references for in-situ and real-time monitoring of film growth by ellipsometry or Raman spectroscopy.

**CP-21 Electrical Properties of Molybdenum Doped  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> Thin Films,** *J Galindo, Anil Krishna Battu, R Chintalapalle*, University of Texas at El Paso, USA

Gallium oxide (Ga<sub>2</sub>O<sub>3</sub>), which is a stable oxide of Ga, has been attracting the scientific and research community in view of its interesting physical, chemical and electrical properties.  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> thin films find numerous applications in high temperature sensors, photovoltaics, optoelectronics, and anti-reflection coatings. The structural and electrical properties  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> thin films are quite important for their integration into optoelectronics, photovoltaics and sensors. Recently, we proposed an approach to obtain tunable structural and electronic properties of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> thin films using refractory metal incorporation. In this work, we performed a comprehensive study of the electrical properties of molybdenum (Mo) doped  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> (GMO) thin films. The results indicate that the resistivity, sheet resistance, conductivity, charge carrier concentration, and mobility are dependent on the microstructure, chemistry and Mo-content. Most importantly, the variable Mo content from 0 to 12 at% found to influence the electrical properties and allow us to obtain GMO films with a wide range of electrical properties. The results and implications for utilizing GMO films in electronic and optoelectronic devices will be discussed.

**CP-22 Electron Beam Deposition and Characterization of Transparent WO<sub>3</sub>/Al/WO<sub>3</sub> Multilayer Thin Films,** *A Leyva, K Makeswaran, Ramana Chintalapalle*, University of Texas at El Paso, USA **INVITED**

The ever-increasing demand for sustainable energy calls upon practices for efficient energy utilization, management, and harvesting. Smart windows, which can control the throughput of visible light and solar radiation in buildings can contribute to the energy efficiency and cost savings. In this work, an attempt is made to fabricate the multilayered D/M/D architecture using WO<sub>3</sub> (D) and Al (M). The effect of Al thickness [Al(t)] on the performance of WO<sub>3</sub>/Al/WO<sub>3</sub> multilayered films fabricated by electron-beam deposition onto silicon and glass substrates has been evaluated. Efforts were made to understand the effect of Al(t) on the structure, morphology, mechanical durability and optical behavior of the D/M/D multilayered films. The optical, electrical and mechanical responses suggest possible integration of these WO<sub>3</sub>/Al/WO<sub>3</sub> films for smart window applications with enhanced performance.

**CP-24 Numerical Ellipsometry: Extension of Concepts of n-k Plane Solutions from Isotropic to Anisotropic Films,** *Frank Urban, D Barton*, Florida International University, USA

Ellipsometry is an optical technique for determining properties of laminar reflecting (or transmitting) structures from the measurement of light polarization state change resulting from the light-sample interaction. In all but the simplest of cases, the measurements must be followed by data processing in which the measured data is compared to an appropriate mathematical model of the sample derived from Maxwell's equations and measurement conditions. Thus a key common problem is determining how many and which measurements to make. A single ellipsometer measurement consists of a single complex data point and therefore usually more than one measurement is required. Previously we have addressed these problems using Complex Analysis in the n-k plane for homogeneous isotropic films. One of the key findings was that certain angles and wavelengths are useful and others are not. The purpose of the work here is to extend that analysis to anisotropic films. These films have a greater number of unknowns and consequently require more measurements. The central question remains, how many measurements are needed and which specific measurements will provide sufficiently independent equations considering the unknowns. It can be that additional measurements vary to such a small degree that they are not useful considering measurement error tolerance. The work to be presented will show how to identify useful measurements based upon the anticipated sample configuration. The approach avoids making unnecessary measurements which can actually reduce rather than increase the solution accuracy.

**CP-25 Piezophotocatalytic and Piezoelectric Performance of Titanium Zinc Nitride Nanorod,** *Hsin-Yi Lee, K Chang*, National Cheng Kung University (NCKU), Taiwan

Flexible strain sensors have many applications such as structural health monitoring, mechanical testing, and pulse power suppliers. Piezotronic strain sensors, which consist of a metal–semiconductor– metal interface,

are well-suited for these applications due to their high sensitivity and fast response times. Zinc oxide (ZnO) nanowires (NWs) are a popular material for use in piezotronic strain sensors.[3] However, Zinc oxide has relatively high work function, so we can enhance its field electron emission with titanium nitride (TiN) coating, which has good electrical conductivity and relatively low work function.[1][4][5] Therefore, TiN thin film makes it potential in ideal field emitters. In our research, we want to develop the new material which has piezo-related properties and low work function simultaneously.

In this work, piezophotocatalytic and piezoelectric performance of Titanium Zinc Nitride Nanorod thin films deposited by RF magnetron sputtering were described. TiN and ZnN have centrosymmetric structure. However, thin film capacitors fabricated by sputtering Zn doped TiN nanorods from Zinc and Titanium targets in N<sub>2</sub> ambient has non-centrosymmetric structure, because electric polarization and relative permittivity measurements yield distinct ferroelectric properties.

Based on various measurements including piezopotential, piezotronic, piezophototronic, and piezophotocatalytic analyses obtained by characterization tools, (i.e. X-ray diffraction, X-ray photoelectron spectroscopy, Raman scattering, Scanning electron microscope, Transmission electron microscopy, Secondary-ion mass spectrometry, UV-Vis, and I-V methods) we found that the base pressure of vacuum chamber, the chamber pressure and temperature, the sputtering power, and gas flow significantly influenced this material's crystallinity, morphology (i.e. surface roughness), structure properties (i.e. crystallite size), electrical properties (i.e. refractive index), optical, and mechanical properties. In addition, we use combinatorial methodology to fabricate the material [6], which has significant piezoelectric properties in the specific concentration of Zinc, for use as a piezoelectric sensor.

Keywords: Titanium Zinc Nitride, Zinc doped, nanocolumn, morphology control, composition spread, combinatorial magnetron sputtering, piezotronic / piezophototronic effects, photocatalysis / piezophotocatalysis.

**CP-26 Well-alignment ZnSnO<sub>3</sub> by Epitaxially Oriented PVDF and Synergistic Piezo-related Performance of the ZnSnO<sub>3</sub>/PVDF Nanocomposites,** *Chen-Hui Chou, K Chang*, National Cheng Kung University (NCKU), Taiwan

According to previous researches, two-step hydrothermal method was used to deposit ZnSnO<sub>3</sub> on the different kinds of substrate and control their alignment with different conditions such as substrate, temperature, surfactant, and others.<sup>[1,2]</sup> In this research, a novel way was proposed to fabricate ZnSnO<sub>3</sub>/polymer nanocomposites by simple hydrothermal and polymer epitaxy method. This research emphasized on improving the alignment of ZnSnO<sub>3</sub> nanorods<sup>[3,4]</sup> by polymer epitaxy<sup>[5]</sup> such as PVDF and its synergistic piezo-related performance of the ZnSnO<sub>3</sub>/PVDF nanocomposites.<sup>[6]</sup> PVDF was used to control the alignment of the fabricated ZnSnO<sub>3</sub> nanorods and enhance its piezo-related performance including piezopotential, piezotronic, piezophototronic, and piezophotocatalytic analyses.

XRD and SEM were used to characterize the ZnSnO<sub>3</sub>/PVDF nanocomposites. The results from the XRD confirmed the presence of ZnSnO<sub>3</sub>. SEM analysis showed the morphologies and alignments of the ZnSnO<sub>3</sub> nanorods and PVDF. These nanocomposites exhibited average piezopotentials. Piezotronic analysis was also conducted on ZnSnO<sub>3</sub>/PVDF nanocomposites, exhibiting high current density when the ZnSnO<sub>3</sub> are well-aligned. When under UV light illumination, the output current density obtained were several times higher for ZnSnO<sub>3</sub>/PVDF. These confirmed the alignment control and synergistic piezophototronic property of the material.

In a piezophotocatalytic experiment, the decomposition of methylene blue (MB) was also investigated. The ZnSnO<sub>3</sub>/PVDF nanocomposites exhibited better degradation property than pure ZnSnO<sub>3</sub>. All the exhibiting enhancement was attributed to the well-aligned ZnSnO<sub>3</sub>, which reduced the recombination of photogenerated electron–hole pairs and enhanced the mobility of these pairs resulting from the energy band distortion caused by applied stresses. Finally, we can use this nanocomposites or this epitaxially fabricating method to other materials on various electronic applications, such as multifunctional electronic-skin.<sup>[7]</sup>

Keywords: ZnSnO<sub>3</sub>/PVDF nanocomposites, epitaxy, ZnSnO<sub>3</sub> nanorods, piezophotocatalysis, electronic-skin

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## CP-27 Challenges and Limitations for the Optical Characterization of Sub-micron Temperature Fields in Plasmonic Metamaterials, Juan Antonio Zapien, City University of Hong Kong, Hong Kong

Surface plasmons at metal-dielectric interfaces can efficiently confine and amplify electromagnetic (EM) energy in deep sub-wavelength volumes. Concomitant with the large EM field enhancement, Joule heating occurs which severely limits performance for applications such as miniaturized optical circuits. However, the same effect provides a great opportunity to remotely control temperature distributions in the micro- to nano- scale. These highly localized thermal fields have applications to research in catalysis, heat-assisted magnetic recording, phononic circuitry, and photothermal medical therapy among others. A fundamental step to advance the emerging applications of thermoplasmonics is the capability to provide fast, quantitative, contactless experimental determination of the resulting temperature distributions.<sup>[i]</sup>

Spectroscopic ellipsometry (SE) is a phase sensitivity and self-referenced technique expected to have tremendous impact for contactless, marker-free, optical characterization at the nano-scale.<sup>[ii]</sup> However, numerical, fully-vectorial SE data analysis is required for non-layered samples with characteristic lateral dimensions (L) between  $L/10$  and  $10(L)$ . To date, two systematic approaches seem favorable and will be reviewed; namely, the rigorous coupled-wave analysis (RCWA) method which was highly successful to study optical critical dimension (OCD) of 1D gratings by the semiconductor industry,<sup>[iii]</sup> and, more recently, the Finite-Difference Time-Domain that is being systematically investigated by our group.<sup>[iv]</sup>

In this presentation we will provide an assessment of the expected capabilities of SE to provide quantitative optical characterization in plasmonic metamaterials including changes in refractive index and thermal expansion effects based on the known instrumentation and computational limitations currently available.

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## CP-28 The Modification of Refractive Index by using Solid State Diffusion, Hung-Pin Chen, W Cho, Instrument Technology Research Center, National Applied Research Laboratories, Taiwan; C Lee, National Central University, Taiwan; Y Lin, National Tsing Hua University, Taiwan; W Chen, Instrument Technology Research Center, Taiwan

The optical coatings with excellence performance would be achieved more easily when the materials chosen have relatively adjustable refractive index. In this study,  $\text{Al}_2\text{O}_3/\text{ZnO}/\text{Al}_2\text{O}_3$  structures were fabricated using electron beam evaporation and 800°C post-annealing treatments were carried out. According to the inter-diffusion, the ZnO layer became a high refractive index material with porous structure and the  $\text{ZnAl}_2\text{O}_4$  spinel was formed as low refractive index material and the refractive index contrast of the multilayer was increased. In the  $\text{Al}_2\text{O}_3/\text{ZnO}/\text{Al}_2\text{O}_3$  structure, the porous ZnO layer with an average porosity of 19.78% was successfully prepared and the refractive index was from 2 reduced to 1.357 by 800°C post-annealing process due to solid state diffusion mechanism.

## CP-29 Effect of Silicon Content on the Structural, Optical and Electrical Characteristics of SiO<sub>x</sub> Films Prepared by Sputtering, Karim Monfil Leyva, A Salazar Valdez, Benemérita Universidad Autónoma de Puebla, Mexico; A Morales Sánchez, Centro de Investigación en Materiales Avanzados SC, Mexico; J Luna López, M Domínguez Jiménez, A Muñoz Zurita, Benemérita Universidad Autónoma de Puebla, Mexico

Currently, electronics and semiconductor studies have focused a great effort to overcome the intrinsic disadvantages of bulk-Si to develop optoelectronic devices. The non-stoichiometric Silicon dioxide ( $\text{SiO}_x$ ) has been proposed as a cheap and effective alternative to develop ultraviolet absorbers or silicon-based light emitters.  $\text{SiO}_x$  can be deposited by several physical vapor deposition techniques but Sputtering technique particularly allows a great control on film thickness.  $\text{SiO}_x$  films can be obtained by simultaneous co-sputtering of Si and fused quartz ( $\text{SiO}_2$ ) targets. The Si content in the  $\text{SiO}_x$  layers can be modified by a variation on RF-power applied to Si (Psi) target and keeping constant the RF-power applied to  $\text{SiO}_2$  target.

In this work, we studied the effect of the increase of silicon content on the optical, structural and electrical properties of thin  $\text{SiO}_x$  films obtained by Sputtering. The Psi was changed between 10 and 50 W. All the films were annealed at 1100 °C in  $\text{N}_2$  for 3 hours. Ellipsometry and step measurements were applied to calculate thickness and the refractive index. Fourier transform infrared (FTIR) measurements were obtained from all the  $\text{SiO}_x$  films to confirm a change on stoichiometry. Absorbance spectra of  $\text{SiO}_x$  films showed rocking and bending vibration modes similar to stoichiometric silicon dioxide but an asymmetric stretching mode revealed the non-stoichiometric nature of our  $\text{SiO}_x$  films. X-ray photoelectron spectroscopy (XPS) measurements in depth profile revealed that Si content was increased from 3.5 to 10.7% in the  $\text{SiO}_x$  films. AFM images were obtained to calculate and relate the surface roughness according to Si content.  $\text{SiO}_x$  films showed a wide photoluminescence (PL) at room temperature (RT) between 575 nm to 875 nm. The PL intensity and peak position also showed a dependence on Silicon content and the possible presence of defects.

An Indium-Zinc Oxide (IZO) gate, optically semitransparent in the visible range, was deposited onto the  $\text{SiO}_x$  films surface. Also, Aluminum backside contact was added by Sputtering. Current-voltage (I-V) measurements of IZO/ $\text{SiO}_x$ /Si devices were obtained. A high conduction regime was measured even for low gate voltages. Electroluminescent (EL) emission was observed with the naked eye as discrete shining points on the surface of the devices. The intensity of the shining points showed a dependence on the Si content of  $\text{SiO}_x$  films. The EL emission was related to the recombination of charge moving through conductive paths within the  $\text{SiO}_x$  film.

## CP-30 Optical Properties of the TiO<sub>2</sub> Films Grown by Atomic Layer Deposition using Tetrakis(Dimethylamino)Titanium and H<sub>2</sub>O, Wen-Hao Cho, P Huang, C Chen, Y Yu, C Yang, C Kei, Instrument Technology Research Center, National Applied Research Laboratories, Taiwan

Titanium dioxide ( $\text{TiO}_2$ ) is a promising material due to its attractive physical and chemical properties. Atomic layer deposition (ALD) is one of the deposition methods for  $\text{TiO}_2$  and could provide films with excellent conformality. The  $\text{TiCl}_4$  precursor has been widely used with  $\text{H}_2\text{O}$  to deposit  $\text{TiO}_2$  films in ALD process. However, the reaction by-product HCl are corrosive. In this study,  $\text{TiO}_2$  films were grown on silicon substrates and glass by ALD using  $\text{H}_2\text{O}$  and tetrakis(dimethylamino)titanium (TDMAT) instead of  $\text{TiCl}_4$  to avoid the damage from reaction by-product. The thickness of  $\text{TiO}_2$  films at different substrate temperature were measured by ellipsometer and the highest growth rates per cycle (GPC) was 0.6649 Å at 100°C. The X-ray diffraction showed that the  $\text{TiO}_2$  films were anatase phase. The transmittance (T) and reflectance (R) were measured and the absorption was obtained from 1-T-R. From absorption spectrum, we found that the  $\text{TiO}_2$  films had slight absorption in visible light range. This should be caused by the residual carbon and nitrogen in the films. Furthermore, the  $\text{TiO}_2$  film had lowest absorption at 100°C. The composition analyses were also carried out.

## CP-31 Fractal Analysis of Titanium Nitride Films with Different Morphologies and Evaluation for the Direct Methanol Fuel Cell Applications, Kai-Ling Chuang, M Tsai, Y Tsai, F Lu, National Chung Hsing University, Taiwan

TiN is an important coating material with many technological applications because of its high hardness and chemical stability, as well as low resistivity. With various surface morphologies, conductive TiN films are expected to take part in many more applications. In our previous work,



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granular and pyramidal morphologies TiN films could be tailored by merely adjusting the gas flow ratio, leading to changes in the texture coefficients. The objective of this work is to further analyze morphologies of the TiN films by estimating fractal dimensions of the films. The TiN films with different morphologies were then employed for the supporting electrodes on direct methanol fuel cells. Pyramidal and granular TiN films were produced with N<sub>2</sub>, air, and simulated-air as the reactive gas during magnetron sputtering. With the TiN films turning from granular to pyramidal morphology, the value of fractal dimension decreased from 1.95 to around 1.63 for those three reactive gases. The development of the morphologies was associated with the texture of the films that changed from rather random orientation to a mainly (111) preferred orientation. From the test of methanol oxidation, the TiN electrodes with different morphologies showed promising current densities responses, compared to traditional metal and carbon supports.

## **CP-32 Growth Kinetics Behavior and Morphology of Multicomponent Coating on Zirconium Hydride during Oxidizing Atmosphere, G Yan, Jiandong Zhang, L Wang, S Bai, GRINM company, China**

Zirconium hydride, as essential structural materials within the nuclear reactors, has the problem of hydrogen loss, which reduces the neutron moderating efficiency and service life. To prevent or slow down the hydrogen loss in moderator from zirconium hydride, multicomponent coatings (MC) for preventing hydrogen escaping were formed on zirconium hydride by in-situ oxidation method in oxidizing atmosphere decomposed by carbamide. Growth kinetics and morphology of in-situ oxidation MC on zirconium hydride were studied using x-ray diffraction (XRD), scanning electron microscope (SEM), auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy(XPS), focusing on kinetics of growth kinetics and morphology of the coating. As a result, it shows growth kinetics followed a parabolic law with respect to in-situ MC duration. XRD shows the phase structure of coating consists mainly of ZrN and ZrO<sub>2</sub>, including tetragonal zirconia (*t*-ZrO<sub>2</sub>) and monoclinic zirconia (*m*-ZrO<sub>2</sub>). SEM depicts coatings were accumulated and combined densely with substrate. AES indicates that the coating is mainly composed of carbon, nitrogen, oxygen and zirconium, which show that the atomic concentration of carbon and nitrogen decreased continuously, while, oxygen and zirconium increased with sputter time increasing. XPS investigates the chemical binding states in the coatings were Zr-O、Zr-C、Zr-N-O、Zr-N bonds. Under the test of simulated working condition, the MC reduce the hydrogen loss effectively, compared with other single oxide/nitride coatings.

## Fundamentals and Technology of Multifunctional Materials and Devices

### Room Sunrise - Session C2-3

#### Novel Oxide Films for Active Devices

**Moderators:** Marko Tadjer, Naval Research Laboratory, USA, Vanya Darakchieva, Linköping University, Sweden

8:00am **C2-3-1 Improved the PI Transmittance and ITO Conductivity by Supercritical CO<sub>2</sub> Fluid Treatment**, *G Chen, ChienYu Lin, T Chang*, National Sun Yat-Sen University, Taiwan; *S Lin, M Yu, Y Chuang*, HannStar Display Corp, Taiwan

In the flexible display, PI is often selected as the substrate, to achieve the requirement of foldable devices. The electrode of the display must be made of transparent metal. In this paper, the PI transmittance and ITO conductivity was improved by supercritical CO<sub>2</sub> (SCCO<sub>2</sub>) fluid technology. The advantages of supercritical fluid treatments are high penetration and high solubility can easily take away impurities and dope the film at room temperature. Supercritical fluid technology can bring out the monomer molecules and eliminate defects in PI substrate, inducing to higher transmittance. Hydrogenation SCCO<sub>2</sub> increase the conductivity of ITO to improve device performance.

8:20am **C2-3-2 Improving Performance by Inserting an In<sub>2</sub>O<sub>3</sub> Layer into HfO<sub>2</sub>-Based Resistive Random Access Memory**, *Cheng-Hsien Wu*, National Sun Yat-Sen University, Taiwan; *S Lin*, National Tsing Hua University, Taiwan; *T Chang, T Tsai, Y Lin, Y Tseng*, National Sun Yat-Sen University, Taiwan

This letter investigates the characteristics of inserting an indium-oxide (In<sub>2</sub>O<sub>3</sub>) layer in HfO<sub>2</sub>-based resistive random access memory (RRAM). Inserting In<sub>2</sub>O<sub>3</sub> layer in the Pt/HfO<sub>2</sub>/TiN structure not only reduces the forming voltage and the operating current, but also enlarges the memory window. The fitting result of the current-voltage (I-V) curves shows that the conduction mechanisms in high resistance state (HRS) are dominated by Schottky emission conduction for both structures. However, in low resistance state (LRS), the conduction mechanism is Ohmic conduction in Pt/HfO<sub>2</sub>/TiN, but Poole-Frenkel emission mechanism in Pt/HfO<sub>2</sub>/In<sub>2</sub>O<sub>3</sub>/TiN. Based on the material analysis and the current fitting result, a physical model is proposed to explain this phenomenon.

8:40am **C2-3-3 Halide Vapor Phase Epitaxy of Ga<sub>2</sub>O<sub>3</sub>**, *Ken Goto, Q Thieu, D Wakimoto, K Sasaki*, Novel Crystal Technology, Inc., Japan; *K Konishi, H Murakami, Y Kumagai*, Tokyo University of Agriculture and Technology, Japan; *A Kuramata*, Novel Crystal Technology, Inc., Japan; *S Yamakoshi*, Tamura Corporation, Inc., Japan

#### INVITED

Gallium oxide (Ga<sub>2</sub>O<sub>3</sub>) has been actively researched and developed as one of the wide-bandgap semiconductors for next-generation power devices in recent years, and literatures on high-voltage operation of SBDs and FETs were reported. However, in order to introduce Ga<sub>2</sub>O<sub>3</sub>-based power devices to the market, it is inevitable to establish mass production technologies, especially manufacturing technology for large-diameter wafers with thick and conductivity-controlled epitaxial films on them. Epitaxial growth of single crystal Ga<sub>2</sub>O<sub>3</sub> films is reported, which is achieved by using several growth methods such as MBE, PLD, CVD (including MOCVD and mist-CVD) and halide vapor phase epitaxy (HVPE). Among them, HVPE method has demonstrated high-speed growth and doping control over a wide range, and it is suitable for a commercial use because it has a simple hardware structure without using a vacuum system, for instance.

It was thermodynamically analyzed that HVPE growth of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> is possible by using gallium mono-chloride (GaCl) and oxygen (O<sub>2</sub>) as precursors, and a high-purity single crystal homoepitaxial film can be grown at a high temperature of 1000 °C in a hot-wall reactor. The growth rate increased in proportion to the input partial pressure of GaCl gas, achieving 20  $\mu\text{m}/\text{h}$  or more. Si doping into the epitaxial film was carried out by simultaneous supply of SiCl<sub>4</sub> gas into the reactor during the growth, and Si concentration in the range of 3E+15 to 1E+18 cm<sup>-3</sup> can be controlled. It was confirmed that the Si-doped films show n-type conductivity and their carrier concentration (measured by Van der Pauw method) equal to Si concentration in the epitaxial films (measured by SIMS analysis). The mobility at room temperature was nearly 150 cm<sup>2</sup>/Vs when carrier concentration was 1E+16 cm<sup>-3</sup>.

By applying those findings, we demonstrated homoepitaxial growth by HVPE on a 2-inch-diameter substrate for the first time. Single crystal film

was grown on the entire surface of the substrate: mean value (uniformity) of the film thickness and the carrier concentration in the 2-inch-diameter wafer were 10.9  $\mu\text{m}$  ( $\pm 16.5\%$ ) and 2.7E+16 cm<sup>-3</sup> ( $\pm 19.7\%$ ), respectively. It was demonstrated that any SBDs fabricated on this wafer worked normally and fluctuation of on-resistances was 3.8-7.7 m $\Omega\text{cm}^2$ . Relatively good uniformity was obtained in an early stage of film growth development on the 2-inch-diameter wafer. By optimizing the gas flow, the film thickness uniformity improved to 8.0 %. Currently, we devote much effort on improving uniformity.

9:20am **C2-3-5 Severe Positive Bias Temperature Instability in N-type MOS Device with Dipole Doped HfO<sub>2</sub> Dielectric Layer**, *FuYuan Jin, T Chang, H Liu, C Lin*, National Sun Yat-Sen University, Taiwan; *J Liao*, National Tsing Hua University, Taiwan; *F Ciou, W Hung*, National Sun Yat-Sen University, Taiwan

In this work, we found severe positive bias temperature instability (PBTI) in N-type MOS device with dipole doped HfO<sub>2</sub> dielectric layer than N-type MOS device with pure HfO<sub>2</sub> dielectric layer. In addition, both electron trapping and defect generation are also more severe in dipole doping device. This phenomenon can be due to the lowering of the conduction band in HfO<sub>2</sub> with higher electric field which is induced by dipole at interface.

9:40am **C2-3-6 Physical Mechanisms of Negative Bias Illumination Stress in InGaZnO Thin Film Transistors with Different Metal Gate Structure**, *Chung-I Yang*, National Chiao Tung University, Taiwan; *T Chang*, National Sun Yat-Sen University, Taiwan; *W Chou*, National Chiao Tung University, Taiwan

In this study, the electrical analyses and physical mechanisms of structure-dependent reliability tests in InGaZnO thin film transistors are investigated. First, the difference of shielded area between IGZO layer and metal gate is discussed. Under the different metal gate length devices, an abnormal rise in capacitance at the off-state in capacitance-voltage characteristics curves can be observed. It is attributed to edge effect-induced high electrical field when the metal gate length is shorter than IGZO layer length. Under light illumination measurement, the behaviors of subthreshold-leakage current can be observed whether the lengths of metal gate are larger than IGZO layer or not. After the negative gate bias illumination stress (NBIS), it is found that the devices which have edge effect caused the more severe hole injection into the gate insulator.

10:00am **C2-3-7 Fabrication of MSM UV Photodetector Based on ZnO/TFMG/UNCD Nanostructures**, *Markos M. Yenesew, B Huang*, National Taiwan University of Science and Technology, Taiwan; *J Chu*, National Taiwan University of Science and Technology (NTUST), Taiwan

In this study, we demonstrate the fabrication of a high performance photodetectors using a multilayer of ZnO nanotubes (NTs), thin film metallic glass (TFMG), and ultra-nano crystalline diamond (UNCD). The device is fabricated by depositing UNCD and TFMG on a glass substrate in microwave plasma enhanced-CVD and RF magnetron sputtering systems respectively. Finally, ZnO NTs are grown by two-step hydrothermal technique. Systematic device performance investigations have shown a high on/off ratio and a fast response speed at 5 V external bias. The developed fabrication design opens up possibility for gas sensor applications.

10:20am **C2-3-8 Effect of Cadmium Chloride Treatment on Poly-crystalline Thin Films of CdTe/Cd-Zn-Te/CdTe Structures**, *Tushar Shimpi, C Reich, K Barth, W Sampath*, Colorado State University, USA

By varying the composition of zinc in the ternary alloy of Cd<sub>(1-x)</sub>Zn<sub>x</sub>Te (Cd-Zn-Te), the band gap can be adjusted. This enables optimizing the Cd-Zn-Te top cell depending upon the number of junctions in the multi-junction solar cell.

In this study, sublimated poly-crystalline thin films of Cd-Zn-Te with a band gap of 1.70 eV was sandwiched in between two 100 nm CdTe films. The overall superstrate structure was glass/tin oxide doped with fluorine/Mg-Zn-O/CdTe seed layer/Cd-Zn-Te/CdTe cap. In the previous studies of 1-micron Cd-Zn-Te films and devices, the well-known CdCl<sub>2</sub> defect passivation treatment caused zinc loss in the film through the formation of volatile ZnCl<sub>2</sub>. The loss of zinc reduced the high band gap thin film of Cd-Zn-Te to a lower band gap CdTe (1.48 eV). The objective of this study was to prevent this stoichiometry change by providing a zinc barrier in the form of CdTe cap prior to treatment. The CdTe seed layer was used to prevent delamination of the Cd-Zn-Te films after the CdCl<sub>2</sub> treatment and have a better band alignment at the front interface. After the passivation treatment, electrodes were deposited, and devices fabricated.

## Friday Morning, April 27, 2018

From the external quantum efficiency graph, the current generated was more than 60% including the optical losses in the wavelength range of 350 nm to 700nm. The band edge did not shift towards the longer wavelength region indicating that the band gap did not significantly change, and zinc loss was prevented from the Cd-Zn-Te thin film. The devices exhibited a rectifying curve in the current density and voltage graph. The line scans and the elemental maps collected from the cross-section viewed under a transmission electron microscope further confirmed that most of zinc was retained in the bulk of Cd-Zn-Te. Some diffusion of zinc was seen in the CdTe seed and capping layer. The chlorine decorating the grain boundaries of Cd-Zn-Te and accumulation at the front interface of Mg-Zn-O/CdTe seed layer, seen in effective CdCl<sub>2</sub> treatment of CdTe films was also observed.

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