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 theorical - practical capacity of absorbing photons in a different emission.

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

A colloidal solution was prepare and multi-deposited on soda lime glass covered with ITO substrate by spin-coating technique and annealed at 120°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_xN 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_xN thin films can crystallized in many phases : Ni_4N crystallizes in cubic phase, Ni_3N in hexagonal phase and Ni_2N in tetragonal phase. Most studies, related to NiN by reactive sputtering, report a mixture of nitrided phases and the change from Ni_4N to Ni_3N can be highlighted by the change in the magnetic behavior from ferromagnetic to paramagnetic. This material have 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_2 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_4N, Ni_3N and Ni_2N) appears for 20% N_2 in the discharge.

XPS and EDX are well correlated and permit us to determine three zones : metallic between 0 and 20% N_2, Ni_4N between 20% and 42% N_2 and finally Ni_3N for higher than 42% N_2. 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 NixN 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_2O-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_2O 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_2O sheet surface due to oxygen plasma occurs. In this paper, we describe the photovoltaic properties for Cu_2O-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_2O sheet from the loading chamber. After pre-sputtering, a p-Cu_2O 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_2O 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_2O 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_2O sheets surface was suppressed. In addition, we also prepared AZO/n-type ZnO/p-type Cu_2O solar cells. The photovoltaic characteristics of the solar cell with the AZO/n-type ZnO/p-type Cu_2O structure improved compared with that of the solar cell with the AZO/p-type Cu_2O 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_2O 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_xWO_3 powders by a solution-based chemical route. The precursors are H_2WO_4 and $CsOH{\cdot}H_2O$, 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_xWO_3 samples are characterized by X-ray diffraction, scanning electron microscopy, differential thermal and thermogravimetric analysis and fourier transform infrared spectroscopy. Next, the Cs_xWO_3 powders are sintered at a fixed temperature for different time and Cs_xWO_3 thin films are deposited by electron beam evaporation from the optimal sintered target. Then, the Cs_xWO_3 films are annealed at different temperatures under Ar and H_2 atmosphere. The effects of annealing on the microstructure, morphology and NIR shielding properties of the Cs_xWO_3 films are discussed.

9:20am **C3-5 ZnO Nano-structures Growth and Investigation***, **Alexander Axelevitch**, 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 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, sitall 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[1)] 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_2O. ZnO films grown on a-plane (11-20) sapphire (a-Al_2O_3) substrates exhibited excellent optical and electronic properties.[2)] 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_2O_3 substrates at a substrate temperature of 773 K for 60 min without a buffer layer. The NO gas pressure was varied in the range $4.4´10^{-3}$ to $1.3´10^{-1}$ 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_x 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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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 ºC), which allows us to use polymers as substrate materials. We have reported the successful growth of the ZnO nanorods (NRs) on the Au/SiO_2/Si(100) substrates by CBD using different Zn precursors, *i.e.* $ZnCl_2$, $Zn(CH_3COO)_2·2H_2O$ and $Zn(NO_3)_2·6H_2O$ (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_2/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 $C_6H_{12}N_4$ (HMT) were used as the CBD solutions. Growth time (t_g) 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_g=60 min, the average diameter and length of the NRs on the GZO/glass substrate increased rapidly with t_g. Above t_g=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_2/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_g=5 min, the compressive stress of 0.9 GPa arose. The compressive stress decreased rapidly with t_g. In the t_g range of 10-15 min, the stress changed from compressive to tensile. After that, the stress was returned to the compressive. Above t_g=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_2/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_g, indicating the improvement of the crystalline quality.

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

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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_2O_3 target, while maintaining a constant power of 100W of $Mg_{0.3}Zn_{0.7}O$ 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 $Al_{1-x}Sc_xN$ Thin Films for Pyroelectric and Piezoelectric Applications**, ***Agnė 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^{2}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 ($Al_{1-x}Sc_xN$) 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 $Al_{0.7}Sc_{0.3}N$ 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 $\geqq 0.12$, 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 µm thickness by using 65 KeV Ag^{2+} ion beam at different fluences varying from 10^{12} to 10^{14} ions/cm^2. 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^2 shows maximum increase as compared to the reference cell.

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