

Tuesday Afternoon Poster Sessions, December 13, 2016

Thin Films

Room Mauka - Session TF-TuP

Thin Films Poster Session

TF-TuP1 P-Type Nitrogen Doped Zinc Oxide Films Prepared By Magnetron Sputtering, *J.Y. Chen*, Ming Chi University of Technology, Taiwan; *H.T. Zhang, G. Zhao, Q. Chen*, Beijing Institute of Graphic Communication, China; *Jyh-Shiarn Cherng*, Ming Chi University of Technology, Taiwan, Republic of China

P-type nitrogen doped zinc oxide (NZO) was prepared on a glass substrate by RF magnetron sputtering using N_2 as the nitrogen source. The structural, electrical and optical properties of the NZO films were investigated by X-ray diffractometry, X-ray photoelectron spectroscopy, Raman spectroscopy, Hall effect and Seebeck effect measurements, and UV-Vis spectroscopy. The electrical properties of the p-type NZO showed a conductivity of $7.6 \times 10^{-2} (\Omega\text{-cm})^{-1}$ and remained stable for over 259 days at the optimized process conditions. With the increase of N_2 flow rate during sputtering, the conductivity of the NZO films exhibited an n-p-n transition. The corresponding mechanism of this transition and the origin of the p-type conductivity were discussed accordingly.

TF-TuP2 Low Temperature Deposition of nc-Silicon Thin Films using SiH_4/H_2 Mixture, *Moniruzzaman Syed*, Lemoyne Owen College, USA; *B. Goh, N. Nazarudin*, University of Malaya, Malaysia; *J. Alam*, University of Memphis; *Y. Hamada*, Lemoyne Owen College, USA; *A. Ali*, King Khalid University, Saudi Arabia

Nanocrystalline-silicon (nc-Si) films were simultaneously deposited on glass and single-crystal Si substrates that were exposed to H_2 plasma excited using RF power = 80 W prior to the film deposition, under 250°C by plasma enhanced chemical vapor deposition using a SiH_4/H_2 mixture. Structural changes of the nc-Si films were investigated by X-ray diffraction, Raman spectroscopy, infrared absorption, UV-VIS and AFM measurements. All nc-Si films were deposited as a function of RF power conditions. $\langle 110 \rangle$ preferentially oriented nc-Si films were observed to grow suddenly with RF power of 80 W resulted in improved crystalline qualities. These results were examined on the basis of the effect of various mechanisms on the crystalline properties, although these mechanisms may jointly determine the properties.

TF-TuP3 Effect of Laser Assisted Local Heating on Mechanical Properties during Indirect Hot Stamping of Tubes, *M.N. Chowdhury, T.T. Nguyen, GiDong Park, T.K.A. Dinh, S.-T. Hong*, University of Ulsan, Republic of Korea; *J.W. Jung, H.N. Han*, Seoul National University, Republic of Korea

Mechanical behaviors of a hot stamping steel tube are experimentally investigated using laser assisted local heating. A partial surface with spiral shape along the length of tube is heated to austenitic temperature by combining the linear movement of the laser and the rotation of the tube. The result of axial crush tests suggests that the laser assisted local heating is capable to enhance mechanical strength and hardness of the tube. A microstructural analysis shows that the laser locally induces a martensitic phase transformation in the heated region along the length of the tube.

TF-TuP4 High Quality ALD of Silicon Nitride Films Via Microwave Plasma, *Kihyun Kim*, Samsung Electronics, Republic of Korea; *J. Provine, P. Schindler, Stephen. Walch, Y. Kim, HyoJin. Kim, F.B. Prinz*, Stanford University, USA

Modern integrated circuit processing for DRAM, flash, and logic devices require silicon nitride (SiN) spacer layers with exacting requirements for chemical, physical, and electrical performance. High quality SiN thin films can be achieved by low pressure chemical vapor deposition (LPCVD) at elevated deposition temperatures (>800°C). However, modern device design rules require low thermal budget (<400°C) for these and other applications.

In this abstract, we demonstrate with a custom built PEALD system utilizing a high power (~1kW) microwave plasma source. Utilizing this system, we can achieve composition with low oxygen, carbon, and hydrogen content similar to what is achieved in higher temperature LPCVD reactions.

The dependence of the SiN film properties on process parameters including plasma power and plasma treatment cycle, are investigated. Specifically, the wet etch rates in dilute hydrofluoric acid ($H_2O:HF$ 200:1) with respect to plasma power and treatment cycle times can be varied because of the effects of radicals and ions generated by the plasma source during deposition. Wet etch rates comparable to LPCVD SiN films can be

demonstrated at significantly reduced temperature by PEALD. X-ray reflectometry (XRR) and X-ray photoemission spectroscopy (XPS) studies show the higher plasma power and extended plasma treatment cycles can vary the density and the composition of SiN films.

TF-TuP5 Homeotropic Liquid Crystal Alignment on Aluminum Oxide Films and its Application for Liquid Crystal Display, *Hong-Gyu Park*, Changwon National University, Republic of Korea; *H.Y. Mun, H.-C. Jeong, J.H. Lee*, Yonsei University, Korea, Republic of Korea; *B.-Y. Oh*, ZeSHTech Co. Ltd., Republic of Korea; *D.-S. Seo*, Yonsei University, Korea, Republic of Korea

Liquid crystal display (LCD) technology is still occupying a strong position in the display industry. The orientational control of liquid crystal (LC) molecules is essential for high-quality LCDs and interactions between LC molecules and solid-substrate surfaces are still significant in both fundamental research and industrial applications. Achieving sophisticated control and uniform alignment of LCs with a regular pretilt angle on the alignment layer is an important step in the manufacture of LC displays (LCDs). Intensive research on the use of organic or inorganic films as LC alignment layers has been conducted to obtain uniform LC alignment using various alignment techniques such as rubbing, ultraviolet (UV) photoalignment technique, nanoimprint lithography, oblique deposition, and ion-beam (IB) irradiation. Among these, the rubbing process has still attracted much attention in terms of simplicity, high cost-effectivity, and reliability. Recently, transparent inorganic materials have been investigated by many research groups as alignment layers, because of their low power consumption achieved via the reduction of LC threshold voltage. In addition, oxide-based inorganic materials are more durable than organic materials and are insensitive to oxygen and water, which slows the degradation of the device performance compared to organic materials.

We previously reported on LC alignments on aluminum oxide (Al_2O_3) films deposited using various techniques including atomic layer deposition (ALD), RF magnetron sputter, and e-beam evaporation via IB irradiation. In this paper, we demonstrate the effects of LC alignment on Al_2O_3 films using a rubbing process. Through a simple technique, uniform and vertical LC alignment was achieved on the Al_2O_3 films. Optical retardations and contact angles were observed on the Al_2O_3 films to measure the effect of the rubbing treatment on Al_2O_3 films. Finally, the electro-optical (EO) characteristics of vertically aligned (VA) cells on Al_2O_3 films were measured.

TF-TuP6 A Pulsed Supersonic Valve System for Researches on Collision-Induced Reactive Thin Film Etching Phenomena, *Masahito Tagawa, K. Yokota, T. Yasukochi*, Kobe University, Japan; *S.K. Chee*, Mechano Transformer Corporation, Japan

A pulsed supersonic valve (PSV) with fast operation capability is essential for many molecular/atomic beam experiments. A reliable PSV is a key device for not only scientific molecular beam experiments, but also engineering applications such as future digital molecular beam epitaxy (MBE) and molecular beam-induced etching. Among these applications, laser-detonation hyperthermal beam source, which has been applied for space environmental effect studies on thin film erosion, requires highly reliable and fast response PSV system. We have developed a fast and reliable piezoelectric-driven PSV system. It consists of conventional low-voltage piezoelectric actuator (max 150V) with a displacement enlargement mechanism (displacement is greater than 0.1mm). These mechanisms are covered by a stainless steel body. Size of PSV is approximately 15 x 10 x 4 cm. Gas is ejected by a 1 mm hole. The poppet clearance is adjustable by the micrometer. The poppet system with a Viton O-ring and Au reflector was specially designed for laser-detonation applications, which requires the focusing 7 J laser pulse near the O-ring. A pressure profile of ejected gas from the nozzle was measured. It is clearly indicated that the PSV is able to operate less than 100 microseconds. It should be mentioned that even faster operation is possible with a high-current PSV driver. It was also confirmed the specification below;

Max. Pressure of supply gas: > 0.8 MPa, Max. Repetition rate: > 50 Hz, Poppet life: > 0.6 million shot (with CO_2 laser irradiation)

One of the unique features of this system is that two PSVs independently eject gas pulses into one hole, such that time-variation of the composition of a gas pulse could be controlled. This capability provides controllability of the dissociation of molecules in a pulsed laser plasma.

Time-of-flight spectra of "thermal" molecular beam generated with this PSV system were measured. It was obvious that the velocity distribution was consistent with the Maxwell-Boltzmann distribution. Also the capability of forming "hyperthermal" atomic beam with laser-detonation for space environmental simulation of thin film erosion problems as well as

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a wide variety of applications on applied surface sciences of this fast and reliable PSV system will also be discussed.

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TF-TuP7 Synthesis of High-quality MoS₂ Atomic Layers with Inorganic Seeding Promoters by Chemical Vapor Deposition, Woonchul Yang, S.H. Choi, Y.J. Kim, Dongguk university, Republic of Korea; K.K. Kim, Dongguk University, Republic of Korea

Molybdenum disulfide (MoS₂) is highlighted due to their unique physical and electrical properties such as ultrathin layered structure, high on/off ratio, and high mobility. The Band gap of layered-semiconductor MoS₂ is changed from 1.2 eV to 1.9 eV depending on the layered thickness. Monolayer MoS₂ with direct band gap is an optimal material to develop novel optoelectronic devices. There are many methods to prepare monolayer MoS₂ such as mechanically, chemically exfoliation, and chemical vapor deposition (CVD) method. Among them, the CVD method is inexpensive and easy to synthesize MoS₂ films with large scale and uniform thickness. In the CVD process, the absorption of vapor phase molybdenum and sulfur on the substrate is a problem for laterally layered growth of MoS₂. Recently, organic aromatic molecules help the nucleation of MoS₂ in the CVD process. However, the organic materials are easily decomposed due to their thermal instability at the growth temperature. Herein, we first report the inorganic seeding promoter including alkali metal ions to grow high quality MoS₂ flakes. The size of the grown flakes was up to 200 nm. The crystalline quality and the thickness of the MoS₂ were confirmed by AFM, Raman, and PL. The difference of typical Raman peaks of E_{2g} and A_{1g} is lower than ~19 cm⁻¹ with full width half maximum values of 3.8 cm⁻¹ and 4.5 cm⁻¹, respectively. This high quality is similar with mechanically exfoliated MoS₂. In addition, the size of the grown MoS₂ flakes can be tuned by concentration of the seeding promoters. The growth process with seeding promoters will be suggested in terms of surface reaction and nucleation of participating atoms and promoters on the surface. Our suggested inorganic seeding promoter will open the way to grow high quality monolayer MoS₂ flakes with scalable size.

TF-TuP8 Effect of Al₂O₃ Passivation Layer on the Stability of Al-IZO Thin-film Transistors, Ji-in Park, Y.S. Lim, M.H. Jang, S.I. Choi, N.G. Hwang, M.S. Yi, Pusan National University, Republic of Korea

We investigated the effect of aluminum oxide (Al₂O₃) passivation layer on the stability of aluminum-indium-zinc oxide (Al-IZO) thin-film transistors (TFTs) under positive bias stress (PBS) and negative bias illumination stress (NBIS). 20-nm thick Al-IZO channel layers were deposited by radio-frequency (RF) magnetron co-sputtering, and then annealed in air for 1h at 250°C. Processing parameters such as the IZO, Al₂O₃ target power (50, 10W) and oxygen partial pressure (13%) were fixed. After depositing the channel layers, 20-nm thick Al₂O₃ passivation layers were deposited by RF magnetron sputtering at room temperature, and annealed again at 250°C.

The TFTs without any passivation were taken as reference devices for comparison (device A). Fig. 1 shows the cross-sectional schematic diagram of Al₂O₃ passivated Al-IZO TFTs (device B). The measured electrical parameters of each device were summarized in Table 1, and it indicates that Al-IZO channel layer is not degraded during the passivation layer deposition by sputtering.

Fig. 2 (a) and (b) show the transfer characteristic curves of the device A and B depending on the stress durations (7200 s) under the positive bias stress (V_{GS}= +20V, V_{DS}= 0V, PBS) in dark, respectively. It can be easily seen that threshold voltage shift (ΔV_{th}) of the device B is much smaller than that of the device A, and indicates that more charge trapping sites (O₂+e⁻→O₂⁻) is created in device A during PBS condition. In other words, Al₂O₃ passivation layer could effectively prevent the oxygen absorption on surface of the Al-IZO film under the influence of gate voltage stress.

To investigate the stability of the TFTs under bias illumination stress, a negative bias stress (V_{GS}= -20V, V_{DS}= 0V, NBS) were repeatedly applied to the device A and B under green light illumination in air for 7200s. The brightness of the green light source was 2047cd/m². Fig. 3 (a) and (b) show the evolution of the transfer characteristic curves of each device for various stress durations under NBIS condition, and it can be concluded that the stability of device B is better than that of device A under NBIS condition. In general, the degradation mechanism of the NBS under illumination of Al-IZO TFTs is dominated by the photo-generated hole trapping states (V_o→V_o²⁺+2e⁻) at gate insulator and/or interface between insulator and channel. In this result, the Al₂O₃ passivation layer could effectively

passivate the defect in the Al-IZO films. We demonstrated that the Al₂O₃ could be an effective passivation layer to suppress O₂ absorption on Al-IZO back channel and decrease photo-excitation on Al-IZO films.

TF-TuP9 Extreme Ultraviolet Emission from Laser-induced O₂+Ar Plasmas Relevance to Ground-based Simulation of Thin Film Etching Reactions in Low Earth Orbit, Kumiko Yokota, T. Ohyabu, J. Ohira, K. Morimoto, M. Tagawa, Kobe University, Japan

The environmental factors in space, for example, ultraviolet (including EUV and VUV wavelength), x-rays, ion and electron radiations, thermal cycling, and neutral species such as atomic oxygen (AO), give serious effects on many thin film materials used in space systems. The incompleteness of the ground-based simulation technology arises problems on the accuracy of assessment of the material survivability in real space environment. It has been reported that FEP Teflon eroded much faster in ground-based facilities than in space. This discrepancy is believed due to the ultraviolet which is a byproduct from the oxygen plasma. A laser detonation AO source, which has widely been used as a ground-based AO environmental simulator, uses high-power CO₂ laser to create laser-induced oxygen plasma. The basic configuration of laser detonation AO beam source is similar to that of EUV light source using xenon gas. However, EUV from laser-detonation AO beam source has not been evaluated.

In this presentation, EUV spectra from laser-induced oxygen (and mixed gas) plasmas were reported. The flat-field EUV spectrometer especially designed for this purpose was equipped to laser-detonation AO beam source, and confirmed the capability to measure EUV spectra from oxygen plasma. Also, the cross relationship between EUV spectra, time-of-flight (TOF) spectra of AO beam and effect of addition of Ar were investigated related to the low altitude orbital applications.

It was clearly observed that EUV emission intensity from 100%Ar plasma was greater than that from 100%O₂ plasma. However, Ar emission was not observed from the laser-sustained plasma consisting of 50%Ar+50%O₂ gas mixture, i.e., EUV lines only originated from oxygen were observed. Emission intensities originated from O increased with Ar content and disappeared in the pure Ar plasma. The EUV emission property was considered to be related to the collisional processes in the mixed plasma. This hypothesis was proved by the TOF spectra, i.e., dissociation reaction of O₂ into AO was promoted by adding Ar in the gas. It was also observed that the EUV emission intensity decreased with increasing the supply gas pressure of the PSV. This could be a practical way to reduce the side effect of EUV on the ground-based AO simulations.

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TF-TuP11 Device Performance of Tungsten doped InZnO Thin Film Transistor depending on Active Layer Thickness, Hyun-Woo Park, D.-Y. Kim, Dongguk university, Republic of Korea; W.-C. Yang, Dongguk University, Republic of Korea; M.-D. Kim, Chungnam National University, Republic of Korea; K.-B. Chung, Dongguk university, Republic of Korea

Tungsten doped InZnO thin film transistors (WIZO-TFTs) were fabricated using by radio frequency (RF) sputtering system as a function of active layer thickness. In order to explain the degradation of the device performance depending on the active layer thickness, we investigated the correlations between the device performance and physical properties including the film density, surface/interface roughness, band edge state below the conduction band, refractive index and composition along the depth direction. The 10 nm-thick WIZO film is showed the highest film density and the flat interface roughness. In addition, increase of the band edge state and thickness of interface layers could be related to the device performance, which resulted in changes of interfacial trap density.

TF-TuP12 Influence of Macro-Confinement on the Thermochromic Transition Temperature of VO₂ Thin Films, Adetayo Adedeji, T. Patel, A.D. Agyapong, Elizabeth City State University, USA

Vanadium dioxide (VO₂) in bulk and thin film forms are well known thermochromic materials. VO₂ switches optically from IR transparent to IR reflecting at a transition temperature of about 68°C (about 150°F). Because of the easily accessible transition temperature, researchers are interested in driving the transition temperature to lower or higher values depending on the intended application. The goal of this research is to investigate the effect of thin film macro-size confinement on the transition temperature of

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VO₂. To achieve this goal, pure vanadium metal was deposited on clean quartz substrate using magnetron sputtering system. Different sizes of copper mesh were used as template for the depositions. The samples were oxidized in 800 mtorr N₂/O₂ gas mixture at 500°C for 4 hours. Surface morphology of the sample and chemical composition were obtained using SEM with EDS. Various grain sizes and orientations were observed at different depth from the quartz-VO₂ interface with aberration corrected Scanning Tunneling Electron Microscope (STEM). Optical transmittance in air ambient was measured as the samples were taken through thermal cycle. It was observed that the transition temperatures of samples deposited through Cu meshes are different from those obtained from plain 2D VO₂ films and films shielded with transparent Ta-Si-N barrier layer.

TF-TuP13 Nitrogen Plasma Treatment of Polytetrafluoroethylene, P. Garcia, C. Rangel, Steven Durrant, UNESP - Sorocaba, Brazil

Cold plasma treatment is well established for the modification of metal, glass and polymers. Treatments of polymers in plasmas of inert gases can alter the degree of cross-linking, release hydrogen, and produce dangling bonds, thus affecting the surface morphology and reactivity. In addition to such effects, treatments in plasmas of gases such as oxygen, carbon tetrafluoride or nitrogen, can cause etching and introduce new elements, such as O, F or N, to the polymer surface. It is known that cold nitrogen plasma treatment of polytetrafluoroethylene (PTFE) can strongly modify its properties; for example, reducing its surface contact angle to zero. There is still much to be learnt, however, about the effect of the system parameters on the modifications produced. In this study, PTFE is treated in cold nitrogen plasmas as a function of the gas pressure, P, RF plasma power, W, and exposure time, t. The variables P, W and t range from zero to 100 mTorr, 200 W, and 30 min. respectively. The induced chemical structural and compositional changes are examined using Infrared spectroscopy with attenuated total reflection (ATR) and Energy dispersive X-ray spectroscopy (EDS). Surface roughness and morphology are examined using Scanning electron microscopy (SEM). Surface contact angles are measured using goniometry.

TF-TuP14 Effects of Photogenerated Carrier Scattering on the Decay Process of Coherent Longitudinal Optical Phonons in an *i*-GaAs/*n*-GaAs Epitaxial Structure Investigated by Terahertz Time-domain Spectroscopy, Hideo Takeuchi, T. Sumioka, M. Nakayama, Osaka City University, Japan

Terahertz (THz) electromagnetic waves are generated by illumination of femtosecond (fs) laser pulses on semiconductor surfaces. The THz wave, therefore, supplies time-domain information on the dynamical phenomena around the surface. In the THz-wave emission from the coherent longitudinal optical (LO) phonons, initial polarization, which corresponds to the LO-phonon amplitude, is launched by a surge current of carriers generated by laser-pulse illumination. Subsequently, the coherent LO phonon, the dynamical polarization, starts oscillating and the THz wave is emitted. Here, we point out the possibility that the surge current scatters the coherent LO phonon in the launch stage. For clarifying the above possibility, we investigated the coherent LO phonon in an *i*-GaAs/*n*-GaAs epitaxial structure, focusing our attention on the decay time.

The sample used was grown by MOVPE. The *i*-GaAs (*n*-GaAs) layer thickness was 100 nm (300 μm). The doping density of the *n*-GaAs layer was 3x10¹⁸ cm⁻³. In the *i*-GaAs/*n*-GaAs structure, a uniform built-in electric field is produced in the *i*-GaAs layer, which induces the initial polarization. The THz wave was measured at room temperature and at the humidity below 10%. We used an optical gating method. The fs laser pulse had a photon energy of 1.55 eV and a pulse duration of 60 fs. The laser beam had a Gaussian shape. The pump powers were varied from 30 to 110 mW.

The THz waveforms show a monocycle signal from the surge current, which is followed by long-lived oscillations due to the coherent LO phonon. The Fourier power spectra of the waveforms show the surge current and coherent LO phonon bands. In addition, two bands, the frequencies of which depend on the pump power corresponding to the photogenerated carrier density, were observed. The frequencies of the latter two bands were evaluated using the Gaussian decomposition, and compared with the dispersion curve of the LO-phonon-plasmon coupled (LOPC) mode. We consider that the THz wave from the coherent LOPC mode is emitted from almost the center position of the Gaussian beam spot on the sample surface because of the presence of sufficient photogenerated carriers forming the plasmon. In contrast, the coherent LO phonon is generated around the beam-spot tail. The waveform shows only the coherent LO phonons in the time-delay range larger than 1.0 ps. The decay time of the coherent LO phonon, which is 3.0 ps at the pump power of 30 mW, is monotonically decreased with increasing the pump power: The decay time

is 1.3 ps at 110 mW. Since the increase in the pump power enhances the surge current, we conclude that the surge current disturbs the coherent LO phonon through the scattering.

TF-TuP16 Effects of Borate Polyester Electrolyte on the Anodizing Behaviors and Electrical Properties of ZrO₂-Coated Al Foil, Kaiqiang Zhang, S.S. Park, Kyungpook National University, Korea, Republic of Korea

ZrO₂-Al₂O₃ composite oxide films are promising dielectric material for future use in aluminum electrolytic capacitors. The films were prepared on etched Al foils by ZrO₂ coating and anodizing. The anodizing of Al foils has been carried out generally in aqueous boric acid solution. But when Al foils are anodized above about 700V, boric acid solution is not suitable as electrolyte due to severe O₂ evolution. In this study, we used borate polyester solution as electrolyte to inhibit the O₂ evolution, and discussed the effects of the borate polyester concentration on anodizing behaviors and electrical properties of the ZrO₂ coated Al foils. The results show that borate polyester electrolyte can effectively inhibit the O₂ evolution during anodizing, and increase the withstanding voltage of the samples. The specific capacitance of the samples decreases with the increasing of borate polyester concentration. Compared to anodization in boric acid electrolyte, the use of borate polyester electrolyte led to increases in the specific capacitance of 25.9 % for 700 V, 15.6 % for 800 V and 8.9 % for 900 V.

TF-TuP17 Selectivity of Plasma Treatment Effects on Wettability Modulation between Silicon, Glass, Polyimide and PMMA, Using Several O₂, CF₄ and CHF₃ Gas Ratios, Shogo Uehara, P. Wood, SAMCO Inc., USA

Surface wettability of materials is influenced by surface energy as well as surface roughness, and the surface energy can be modulated by changing surface chemistries. Plasma treatment is one of the techniques to control the surface chemistries, and is used for the surface wettability modulation of materials in device fabrication. However, few papers have investigated the selectivity of plasma treatment effects on the wettability modulation between two or more materials in a batch process [1], [2]. In this research, the roles of reactive species of oxygen- and fluorine-based plasma chemistries were investigated for selective surface wettability modulation of materials. Using a reactive ion etching system (RIE-10NR, SAMCO Inc.), various plasma chemistries with several gas flow ratios of O₂, CF₄ and CHF₃ were formed over inorganic (silicon and glass) and organic (polyimide and PMMA) materials. Static contact angle and XPS were utilized to study the surface wettability and surface chemical bonding structure before and after plasma treatment. The contact angle was also investigated over the course of 20+ days storage in a N₂-purged desiccator in order to examine the stability of the wettability modulation effects.

With a gas flow ratio adjustment, a large contact angle contrast was observed between the materials. In pure CF₄ plasma treatment, the inorganic materials showed contact angle reduction after the plasma treatment, while the organic materials showed significant contact angle increase. XPS analysis revealed that the silicon surface retained 9.5 at.% fluorine, while 35.4 at.% fluorine was observed on PMMA surface after the pure CF₄ plasma treatment. This indicates that the PMMA wettability modulation was caused by the fluorine-containing chemical functional groups at the outermost surface layers.

After ageing the samples in storage, the contact angle contrast was gradually decreased due to a contact angle increase of the inorganic materials after pure O₂ or CF₄ plasma treatment. On the other hand, samples processed in a plasma using a gas mixture of CF₄ and CHF₃ showed stable contact angles. This difference is attributed to the existence of a thin fluorocarbon layer deposited during the CHF₃ based plasma treatment.

These results indicate that the reaction of oxygen and fluorine-containing active species on substrate surfaces influences wettability modulation effects of materials and also the stability of these effects.

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TF-TuP18 Annealing Behaviors for High-k ZrO₂ Grown on Ge, Deuk-Young Kim, W.-C. Yang, K.-B. Chung, S. Lee, Dongguk University, Republic of Korea; M.-D. Kim, Chungnam National University, Republic of Korea; H. Lee, N. An, Y. Lee, Dongguk University, Republic of Korea

The thermal annealing behaviors of electrostatic properties for high-k ZrO₂ layers were investigated. ZrO₂ layers were grown on p-type Ge substrates at 500°C by RF magnetron sputtering. To reduce the native defect density in ZrO₂, we adopted the higher oxygen-partial pressure (*i.e.*, O₂/Ar > 1). The RF power was 150 W, and the growth time was 20 min. After the growth of

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ZrO₂, the samples were annealed at 600-700°C in Ar ambient by using a rapid thermal annealing method. For the capacitance-voltage characteristics, the as-grown sample exhibited its maximum electrostatic capacitance (*i.e.*, C_{max} at the accumulation mode) to drastically decrease with increasing the frequency of ac signals. However, the dielectric characteristics of ZrO₂ were much improved after thermal annealing at temperatures above 600°C. Particularly, the magnitude of C_{max} was increased by more than 3-times after annealing at 600°C. In addition, C_{max} was much stabilized at wide frequency ranges. For higher annealing temperatures, the above feature became more significant. This indicates that the defects in ZrO₂ could be effectively eliminated by thermal annealing; particularly, at temperatures above 600°C. Through x-ray photoelectron spectroscopy measurements, we confirmed the above thermal-annealing behaviors to arise from the stabilization of Zr-O bonds due to the interdiffusion of Ge at the interface between ZrO₂ and Ge. The best electrostatic permittivity was more than 20, and the equivalent-oxide-thickness was less than 4 nm. Owing to the improvement of interface bonding structures at ZrO₂/Ge, the dielectric breakdown field was increased up to ~1 MV/cm. The results suggest that sputter-grown high-k dielectric ZrO₂ can be utilized for Ge-based MOS devices, and that thermal annealing is effective to improve the dielectric characteristics of ZrO₂.

TF-TuP19 Surface Energy Controlled Patterning of Carbon Nanomaterials for Electronic Devices, *SungMin Lee*, S.G. Yoon, Chung Ang university, Republic of Korea

Comparing with traditional coating technique, solution-based patterning method has its strength in process cost as it can be proceeded in room temperature and pressure condition. Because of its merits, solution-based patterning method has been actively exploited in various application such as field effect transistor, flexible transparent electrode, solar cell. However there are some drawbacks in these techniques: controllability of pattern thickness, high material consumption, extended process time. In our study, we design high speed, low cost, thickness controllable, solution-based patterning technique by controlling surface energy of substrate and demonstrate its application to electronic device. Furthermore we also study application for deposition of nano particles. Solution based metal deposition process is very competitive to currently used method vacuum based deposition in point of cost and processing time.

TF-TuP20 Physicochemistry of Chemical Bath Deposited ZnS Films at Near Ambient Temperatures, *Irving Gonzalez*, *I. Oliva Arias*, Centro de Investigación y de Estudios Avanzados del Instituto Politécnico Nacional, Unidad Mérida, Mexico

Zinc sulfide (ZnS) is a semiconductor material that has increased its importance in recent years due to its wide range of applications. For solar applications, ZnS has shown great potential as optical window for developing high efficiency CIGS solar cells. Chemical bath deposition (CBD) technique has been used for several years to obtain ZnS thin films. Deposition time, bath temperature and chemical reagents concentration are some parameters that have been investigated through time. However, there are scarce reports conducted to understand the physicochemistry of the CBD technique. In recent years, the development of physicochemical tools such as the species distribution diagrams (SDDs) and the solubility curves (SCs) gives light to understand the growth mechanisms to obtain the growth parameters for depositing high quality ZnS films. Nevertheless, most of these contributions have been carried out at temperatures between 60 and 90 °C leaving aside near ambient temperatures. Research on these conditions could bring benefits for industrial applications. This work aims to provide the physicochemical conditions in which high quality ZnS films at near ambient temperature can be obtained. A chemical bath containing ZnCl₂, KOH, NH₄NO₃, and SC(NH₂)₂ as chemical reagents was used for depositing the ZnS films. Temperatures of 25, 40, and 55 °C were selected for films deposition. The SDDs and SCs were obtained for each temperature at three conditions (by varying [SC(NH₂)₂]) in order to study the influence of the chemical species onto the ZnS formation. The $R=[HS^-]/[Zn(OH)_2^{2-}]$ ratio as a main parameter for CDB-ZnS films deposition was investigated. The SCs show that higher values of R assure enough amount of HS⁻ ions and better conditions for depositing ZnS, avoiding the formation of Zn(OH)₂. The SDDs show that at near ambient temperatures the ZnS formation is attributed to the Zn(OH)₃⁻ and Zn(OH)₄²⁻ complexes and the HS⁻ ions.

ZnS films deposited onto glass substrates were characterized by x-ray photoelectron spectroscopy, atomic force microscopy, x-ray diffraction, and spectrophotometry techniques. Results indicate that when the R ratio and temperature are increased, better stoichiometry is obtained. The Zn is

bonded mainly to hydroxide ions for lower R ratios, but when R increases bonding changes to S ions. The band gap energy of films was measured between 3.4 and 3.7 eV. The lower values are due to that samples contained ZnS and Zn(OH)₂ because of the low value of R. ZnS films deposited at 55 °C presents a (111) cubic orientation (sphalerita); meanwhile, films deposited at 25 °C present an orthorhombic orientation due to the Zn(OH)₂ formation.

TF-TuP22 Improvement of Detection Limit for Hydrogen by High-resolution ERDA, *Daiichiro Sekiba*, *I. Harayama*, *T. Tamura*, *Y. Watahiki*, *K. Tsujita*, University of Tsukuba, Japan

Recently high-resolution ERDA (Elastic Recoil Detection Analysis) is installed in UTTAC (University of Tsukuba, Tandem Accelerator Complex) in Japan, which consists of a 90 degree magnetic sector lens and a position sensitive detector (PSD). High-resolution ERDA is one of the powerful tools to obtain the depth profile of hydrogen with the depth resolution of sub-nanometer, while its detection limit for hydrogen has not been good enough to discuss the hydrogen concentration at interfaces made on semiconductor substrates. The main origins of noise, which makes the detection limit worse, are dark current in the micro-channel plate (MCP) used as PSD and stray particles in the vacuum chambers. We found out that the dark current can be distinguished from the desirable signal by a coincidence measurement using two MCPs. The two MCPs detect the recoil of hydrogen ion and secondary electrons from stopper foil, respectively. The dark current noise can be suppressed under the 1% to take the coincidence not only of the detection time but also the positions on the two MCP. The second coincidence can remove the chance coincidence of two dark current signals. We have also tried to develop a new PSD based on solid state detector (SSD) usually made by silicon. In this case the PSD has the energy resolution of detected particle, so that, we can distinguish also the stray particles. We report the recent achievement using these new detectors.

TF-TuP23 Investigation of the Low Temperature Formation of DLC Thin Film by Ion Assistance, *Tsuyoshi Inoue*, *I. Takano*, Kogakuin University, Japan

DLC is amorphous carbon that contains a significant fraction of sp² and sp³ bonds. It is known that their films show a low friction coefficient and its high hardness is applied in a mechanical field such as cutting tools or frictional parts. DLC films were formed by the ion beam assisted evaporation method in the early 1970's, and after that have been manufactured by various methods. The film formation of DLC is performed in a high temperature basically. On the contrary the film formation in the negative temperature is hardly performed. In our experiment, an N₂⁺ ion beam to the substrate was irradiated with introduction of a hydrocarbon gas in a vacuum chamber. This deposition method has some independent parameters from the film formation condition in comparison with other dry process methods. Therefore this method is anticipated in appearance of superior characteristics such as a high adhesion. In our previous research, iced water was used to urge adsorption of a hydrocarbon gas on the substrate temperature control. It was suggested that adsorption of a hydrocarbon gas depended on the substrate temperature.

In this study, the control of a substrate temperature was performed by using the LN₂ and heater controlled by PID. Stainless steels (304SS), Si plates and slide glasses were prepared for sample substrates. The formation conditions of DLC films were changed from -150 degrees to 25 degrees and were an N₂ gas pressure of 4x10⁻³ Pa under an ultimate pressure of 6x10⁻⁴ Pa. C₇H₈ as an ambient gas was introduced by to determine the gas ratio using QMS. The ion beam irradiation was performed with 1 kV in an accelerating voltage and 30 μA/cm² in a current density, through an exposure time of 3600 s. The film structure was determined by Raman spectroscopy and the thickness was measured using a spectrometer of a fiber injection type. Vickers hardness and the friction coefficient were measured by a micro hardness tester and a tribotester of a ball-on-disk type, respectively. The tribotester was set with an Si₃N₄ ball as a counter material and the examination was carried out with a constant load of 0.98 N until a sliding distance of 100 m.

I_b/I_g ratios of DLC thin films measured by Raman spectroscopy showed the higher sp³ contents at -100°C of a cooling substrate temperature. The suitable mechanical property of the DLC films was obtained by a temperature of -100 degrees and the friction coefficient was 0.20 at a distance of 100 m.

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TF-TuP24 Understanding the Effect of Nitrogen Plasma PulseTime on Plasma Assisted Atomic Layer Epitaxy of InN Monitored by Real Time GISAXS, *N. Nepal, V.R. Anderson, S.D. Johnson, D.J. Meyer, B.P. Downey*, U.S. Naval Research Laboratory, USA; *Z.R. Robinson*, The College at Brockport SUNY, USA; *K.F. Ludwig*, Boston University, USA; **Charles Eddy, Jr.**, U.S. Naval Research Laboratory, USA

A challenge to the development of III-N semiconductors (AlN, GaN and InN and their ternaries and quaternaries) is the requisite heteroepitaxy on foreign substrates, compounded by increasing device structure complexity. To address ternary layer miscibility gaps, substrate versatility and low strain heterojunctions the growth temperature must be reduced. Recently, plasma assisted atomic layer epitaxy (PA-ALE) has been used to grow InN and In containing ternaries at temperatures ≤ 260 °C [1-2]. At these growth temperatures, ad-atom mobility is low and understanding the mechanism of nucleation and growth is critical for improved material quality. Traditional ultra-high vacuum *in situ* monitoring methods are impractical as the growth pressure for PA-ALE is too high (1-500 mTorr). High intensity coherent x-rays, such as from a synchrotron light source, offer an effective alternative to study PA-ALE nucleation and growth process.

Here we present *in situ* studies of the effect of nitrogen plasma pulse time on temporal evolution of the growth surface for InN growth on a-plane sapphire at 250 °C as revealed by real-time grazing incidence small angle x-ray scattering (GISAXS) measurements. *Ex situ* atomic force microscopy, x-ray photoelectron spectroscopy and reflectivity, and Hall measurements were employed to characterize the resulting films.

GISAXS measurements show that H₂ plasma cleaning roughens the substrate surface, but this same surface is recovered during subsequent N₂ plasma treatment. During the initial cycles of growth the diffuse specular reflection broadens and correlated peaks (CPs) evolve along the native wing with different correlated length scales (CLSs). For a nitrogen plasma pulse time (t_p) of 15 seconds, CPs have two different CLSs of 33.36 and 8.38 nm. With increasing t_p to ≥ 20 s, a single CP evolves and the longest CLS of 11.22 nm is for $t_p = 25$ s. Additionally at $t_p = 25$ s, the growth rate is largest (0.035nm/cycle – consistent with self-limited growth [1]) with root mean square surface roughness and carbon impurity at or below instrument sensitivity limits. Thus the nature of GISAXS CP CLS directly correlate with the material quality. For the $t_p = 25$ s film, measurements show electron sheet carrier density and resistance of 3.5×10^{13} cm⁻² and 3.59 kW/sq, respectively. An electron mobility of 50 cm²/V-s is measured for a 5.6 nm thick InN film, which is higher than the reported value of 30 cm²/V-s for a 1300 nm thick InN grown by molecular beam epitaxy directly on sapphire [3].

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TF-TuP25 Adsorption Isotherms and the Mean Residence Time of Hydrogen Physisorbed on a Copper Surface, *Yuki Kato*, Gakushuin University, Japan; *K. Kawahara, H. Yamazaki, K. Yamakawa, I. Arakawa*, Gakushuin University

In order to achieve ultimate pressure in extreme high vacuum (XHV) region, it is necessary to exhaust hydrogen which is the dominant residual gas in XHV. Cryopumping has been regarded as an effective mean to exhaust hydrogen and widely used in a variety of vacuum systems. Several studies have reported unexpected outcomes in either the static or the dynamic characteristics of H₂ cryopumping, which is likely due to incident thermal radiation, the diffusion of H₂ in porous adsorbents, or the lack of equilibrium of the H₂ distribution on a surface [1,2,3], but its origin has not been clarified yet. The adsorption isotherms measured in the pressure range below 10⁻⁸ Pa were far from being sufficient for quantitative analysis. This is mainly due to the difficulty in determining the density of H₂ physisorbed on a cold surface. In the present study, we utilized electron stimulated desorption (ESD) and time-of-flight (TOF) techniques to determine the density of H₂ physisorbed on a copper surface [4]. The ejection of H⁺ is mainly caused by the dissociative ionization of the H₂ molecule physisorbed. We assume that, in submonolayer range, the ESD yield of H⁺ ions is proportional to the surface density of physisorbed H₂. Using the above methods, we measured the adsorption isotherms of submonolayer H₂ at equilibrium pressures between 10⁻¹⁰ and 10⁻⁶ Pa in the temperature range of 3.8 - 6.5 K. By monitoring the time development of the H⁺ ESD yield in the transition state approaching adsorption equilibrium, we also determined the mean residence time of physisorbed H₂ at various temperatures. One example of the transition measurements, where the initial H₂ coverage is less than 0.001 (the equilibrium pressure as low as

6 $\times 10^{-11}$ Pa) and the final coverage is 0.3 which is in equilibrium with the pressure of 3 $\times 10^{-8}$ Pa, gives the mean residence time of 4400 s.

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TF-TuP26 Far- and Mid-infrared Spectroscopy of Amorphous H₂O Ice Films formed by Matrix Sublimation Method, *Natsumi Suzuki, G. Shimizu, H. Nasu, R. Tsuboi, K. Yamakawa, I. Arakawa*, Gakushuin University, Japan

To reveal the nature of the hydrogen bond is important in understanding the physical properties of gaseous, liquid and solid water. Since water clusters are the simple hydrogen bond systems, our group has investigated their structures and vibrational states in a previous study using infrared absorption spectroscopy combined with the matrix isolation technique.¹ We adopted CH₄ as the matrix species because its infrared active property enables us to investigate the structure of the matrix itself. In a recent study, Kouchi et al. investigated amorphous ice (ASW) formed by the matrix sublimation method showing that the ice was different in structure from vapor-deposited ASW.² Using an apparatus we have constructed for the purpose of spectroscopy in the range from 100 to 600 cm⁻¹ under ultrahigh vacuum, we measured far-infrared spectra of matrix-sublimated ice (MSI) in addition to those in the mid-infrared region.

We used two independent apparatuses for far- and mid-infrared spectroscopy. Each apparatus was equipped with a continuous-flow helium cryostat on which the gold substrate is held by a copper holder and was evacuated to ultrahigh vacuum condition. A gas mixture of CH₄ and H₂O was deposited onto the substrate at 11 K. Spectra were recorded in the reflection configuration with a liquid-helium-cooled silicon bolometer for the far-infrared region and a HgCdTe detector for the mid-infrared one.

In the mid infrared spectrum at 10 K, we observed a sharp peak at 3010 cm⁻¹ caused by the ν_3 vibrational mode of CH₄ and a broad band from 3080 to 3600 cm⁻¹ assigned to bonded-OH stretching of ASW. As the temperature was increased, the CH₄ peak decreased and the ASW band grew. At 60 K, where CH₄ was completely desorbed from the substrate, the broad band changed in structure and showed a characteristic plateau extending from 3080 to 3600cm⁻¹, which implies the formation of MSI. In the far-infrared region, a broad band ranging from 140 to 310 cm⁻¹ also appeared at 60 K. In addition, these spectral features were almost invariant until 115 K. The drastic changes of the spectra corresponding the translation from ASW to crystalline ice were observed in both the mid- and far-infrared regions at 140 K. Comparing these spectra with those of vapor-deposited ASW in detail, we will discuss the structure of MSI.

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TF-TuP28 The Importance of the Empirical Modeling of the Background Signal in XPS Data for Calculating the Composition of Transition Metal Compounds, *Alberto Herrera-Gomez*, CINVESTAV-Unidad Queretaro, Mexico

The increasing scope of transition metal compounds in nanofilm and nanoparticle technology invigorates the need for an accurate characterization of these materials with X-ray photoelectron spectroscopy (XPS). It is interesting to notice that, to the knowledge of the authors, there is not a single published report about the peak fitting of, e.g., metallic iron or metallic zinc. It was not but until recently that the first calculation, through XPS, of the composition of iron¹ and cobalt² oxides was reported. The scarcity of this type of reports is related to the analytical difficulty associated to fitting transition-metal photoemission peaks: they present a steeply rising background³ and a complex multiplet and satellite structure.⁴ In this paper we will describe the analysis tools required for modeling complex background and line-shapes appropriate for composition analysis from XPS data.

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TF-TuP29 Anlnsys On Transport-Current Characteristics According To The Material Properties Of The Outer Layer Of RebcO Thin-Film Superconducting Wire Having Composite Structure, Ho-Ik Du, S.C. Yang, H.G. Jeong, Chonbuk National University, Republic of Korea

To protect REBCO layers that are weak against thermal impact after quenching, REBCO thin-film superconducting wires are manufactured with a complex structure of a substrate layer (metal), a superconducting layer (ceramic), and a stabilizing layer (metal). The stabilizing layer is positioned at the outermost layer of the REBCO thin-film superconducting wire to cover the REBCO superconducting layer, and is made mainly of Ag. The stabilizing layer normally protects the REBCO superconducting layer from thermal stress.

Therefore, in this paper, a REBCO thin film superconducting wire was fabricated by depositing materials with different specific resistance values (Ag) on REBCO thin-film superconducting wire, using the "RF Sputtering Deposition Method" with micro-range thicknesses to form a outer layer. Then the fabricated REBCO thin film superconducting wire were subjected to basic characteristics tests (measurement of their temperature distribution according to their changing resistance) and over-current transport- current tests to investigate their phase transition. Finally, the results of the basic characteristics tests and the over-current transport-current tests were analyzed to present the applications of superconducting power application devices of the REBCO thin film superconducting wire according to the thickness and properties of the wire's stabilization layer

TF-TuP30 Electric Field Strength in Vapor Deposited CO Films determined from Induced Infrared Absorption of H₂, Koichiro Yamakawa, A. Ishibashi, I. Arakawa, Gakushuin University, Japan

Field and co-workers recently reported that the strong electric field, whose strength may exceed 10^8 V/m, exists in a vacuum-deposited film of dipolar molecules and attributed it to the spontaneous alignment of the molecular axes [1-4]. They have utilized two experimental techniques: the direct measurement of the electrostatic potential at the film surface for N₂O [1] and reflection-absorption infrared spectroscopy for N₂O and CO [2-4]. In the latter method, the infrared absorption band due to the intramolecular stretching vibration split into two peaks owing to the coupling with phonons in the film, so that the temperature dependence of the split was attributed to the intense electric field. In order to judge the validity of their argument, one needs experimental data obtained by other techniques as well as theoretical models to explain the mechanism where such thermodynamically unstable film structure is realized simply after vapor deposition onto a cold substrate. In the present study, we directly determine the strength of the electric field in CO films using induced infrared absorption due to H₂.

A sample chamber equipped with a liquid-helium continuous-flow cryostat was in the ultra-high vacuum condition after baking at 373 K for 24 hours. The substrate was a gold plate mechanically fixed on a copper block placed at the end of the cryostat. Temperatures of the substrate were measured with a silicon diode. We prepared two kinds of samples on the substrate maintained at 5 K; I) CO and H₂ were premixed in a gas handling with the molar ratio of CO/ H₂ = 10 and were deposited; II) We condensed two gasses alternatively through different variable leak valves to form their periodic films, where the thickness of each CO (H₂) film was ~40 (1) monolayer. Fourier transform infrared spectroscopy was performed with a HgCdTe detector in the reflection configuration.

For both the kinds of samples, infrared spectra showed a strong absorption peak at 2142 cm⁻¹ due to the CO stretch along with satellite features at 2047 and 2092 cm⁻¹. In addition, weak absorption peaks were found in the H₂ stretch region. We will give detailed assignment of these peaks and will determine the strength of the electric field in each film from the infrared absorption intensity of the H₂ stretch. Temperature dependence of the field strength will be also discussed.

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