Monday Afternoon, November 7, 2016

Thin Film

Room 102B - Session TF+PS+SE-MoA

Plasma-based Deposition Techniques and Film Characterization

Moderators: Jim Fitz-Gerald, University of Virginia, Tansel Karabacak, University of Arkansas at Little Rock

1:40pm **TF+PS+SE-MoA1 Microcrystalline Silicon Thin Film Deposited by Tailored Voltage Waveform Plasmas using an SiF₄/H₂/Ar Chemistry and its Application to Photovoltaics**, *Junkang Wang*, LPICM, CNRS, École Polytechnique, Université Paris Saclay, France; *M. Elyaakoubi*, TFSC-Instrument, Palaiseau, France; *E.V. Johnson*, LPICM, CNRS, École Polytechnique, Université Paris Saclay, France

For the growth of hydrogenated microcrystalline silicon (μ c-Si:H) thin film by low temperature plasma-enhanced chemical vapor deposition (PECVD), silicon tetrafluoride (SiF4) has recently attracted interest as a precursor due to the resilient optoelectronic performance of the resulting material and solar cell device. However, many questions remain concerning the critical factors determining the quality of the PECVD-deposited film.

Tailored voltage waveforms (TVWs), non-sinusoidal voltage waveforms used to excite radio-frequency capacitively coupled plasma (RF-CCP) processes, has recently been shown to be effective to separately control the maximum ion bombardment energy (IBE) and the ion flux on each electrode. Due to this unique feature, TVWs have attracted considerable research interest in a very short time. When applied to the growth of µc-Si:H film by PECVD, it can provide an elegant approach for one to gain more insight into the physical principles governing film growth and the optimization of process parameters.

To advance knowledge on this subject, we present studies looking at the deposition of μ c-Si:H film from SiF₄ using TVWs, particularly focusing on the material's optoelectronic properties and its resulting PIN solar cell device. We underline recently obtained results concerning critical experimental findings: (1) the significant impact of the maximum IBE to the crystalline grains sizes of the deposited films, (2) the considerable different in films' properties resulting from two types of "sawtooth" waveforms, i.e. "sawtooth-up" and "sawtooth-down", which give similar films deposition rates and the maximum IBE but opposite plasma sheath dynamics during processing. The films generated in these studies have furthermore been characterized using the steady-state photoconductivity and steady-state photocarrier grating techniques, analyzing the coplanar electronic transport properties of the material. The modulated photoconductivity method is also utilized to reveal more specific details about the materials' sub-gap density of states. These studies - along with residual gas analysis studies and Fourier transform infrared absorption results - allow us to optimize the appropriate process parameters of such film and its resulting PIN solar cell device using SiF₄ as the precursor.

2:00pm **TF+PS+SE-MoA2 Boron Carbide-Aromatic Composite Films by PECVD: A Novel Approach to Electron-hole Separation**, *B. Dong, A. Oyelade*, University of North Texas; *E.M. Echeverria*, University of Nebraska-Lincoln; *Y-S. Jun, G.D. Stucky*, University of California at Santa Barbara; *P.A. Dowben*, University of Nebraska-Lincoln; *Jeffry Kelber*, University of North Texas

Many photovoltaic and photocatalytic systems employ band-bending at surfaces or interfaces to achieve electron-hole separation and functionality. Boron carbide-aromatic composites, formed by plasmaenhanced co-deposition of carboranes and aromatic precursors, present an alternative approach where such separation is achieved by aromatic coordination to the carborane icosahedra. Photoemission, density functional theory calculations, and variable angle spectroscopic ellipsometry demonstrate that for orthocarborane/pyridine and orthocarborane/aniline films, with controlled aromatic/orthocarborane ratios between 1:1 and 10: 1, states near the valence band maximum are aromatic in character, while states near the conduction band minimum include those of either carborane or aromatic character. Thus, excitation across the band gap results in electrons and holes on carboranes and aromatics, respectively. Further such aromatic-carborane interaction dramatically shrinks the indirect band gap from 3 eV (PECVD orthocarborane) to ~ 1.6 eV (PECVD orthocarborane/pyridine) to ~1.0 eV (PECVD orthocarborane/aniline), with little variation in such properties with aromatic/orthocarborane stoichiometry. Recent photoabsorbance measurements show that in orthocarborane/pyridine films, the indirect band gap energy is significantly less than the exciton formation energy of 2.1 eV, allowing facile exciton elimination by phonon scattering of electrons into the conduction band at room temperature. The opposite is true for the PECVD orthocarborane film, where the exciton formation energy (2.4 eV) is less than the indirect band gap, inhibiting exciton elimination by electron-hole separation. The enhanced electron-hole separation, narrowed band gap, and significantly increased carrier lifetimes (350 µsec for PECVD orthocarborane/pyridine vs 35 µsec for PECVD orthocarborane), indicate the potential for greatly enhanced charge generation, as confirmed by zero-bias neutron voltaic studies. Those results--an 850% increase in charge generation per B atom for the PECVD pyridine/orthocarborane film relative to the PECVD orthocarborane film-indicate that the enhanced electron-hole separation and band gap narrowing observed for aromatic/orthocarborane films relative to PECVD orthocarborane, have significant potential for a range of applications, including neutron detection, photovoltaics, and photocatalysis.

Acknowledgements: This work was supported by the Defense Threat Reduction Agency (Grant No. HDTRA1-14-1-0041). The authors would like to thank Shireen Adenwalla for technical assistance and discussion. James Hilfiker is also gratefully acknowledged for stimulating discussions.

2:20pm TF+PS+SE-MoA3 Impact of Pulsing the rf Power and the Precursor Injection on the Structure and Optical Properties of TiO₂ and TiSiO Thin Films Deposited by PECVD, Agnes Granier, S. Elisabeth, R. Michaud, N. Gautier, M. Richard Plouet, IMN, University of Nantes CNRS, France; M. Carette, IEMN CNRS/Université Lille 1, France; A. Goullet, IMN, University of Nantes CNRS, France

TiO₂ thin films are good candidates for the development of passive optical components due to high optical refractive index (1.8 < n < 2.7 at 633 nm) combined with high transparency in the visible range. They are compatible with semiconductor technologies and can be synthesized at low temperature by plasma enhanced chemical vapor deposition (PECVD). PECVD is known for its ability to prepare amorphous or partially crystallized films at low temperature and to tune the film composition and optical properties. In the case of TiO₂, columnar polycrystalline anatase films can be prepared by PECVD at substrate temperature less than 150°C. Whereas these TiO2 films are very attractive for photocatalysis, their columnar structure and low optical gap (3.2 eV) appear to be drawbacks for optical applications. Adding a small amount of silicon to TiO₂ allows both obtaining amorphous films and increasing the optical gap, which is highly suitable for optical applications.

In this study, TiO₂ and Ti-Si-O films were deposited in a low pressure rf inductively coupled plasma (ICP) from titanium tetraisopropoxide (TTIP - Ti(OC₃H₇)₄) and hexamethyldisiloxane (HMDSO - SiO₂(CH₃)₆) vapors mixed with oxygen. The structure and chemical composition of the films were investigated by X-ray diffraction, photoelectron spectroscopy, Fourier transform infrared spectroscopy and Raman spectroscopy. The morphology of the thin films was characterized by scanning and transmission electron microscopies. The optical properties were investigated by UV -Visible spectroscopic ellipsometry and absorption spectroscopy. When deposited at the floating potential, the TiO₂ films deposited in the continuous mode in oxygen rich O₂/TTIP ICP plasmas were previously shown to be columnar and highly crystallized in the anatase form. As silicon is added to titanium, the films become amorphous. Their refractive index decreases and their optical gap increases [1].

Here, we investigate the effects of pulsing both the rf power and the precursor injection on the film structure and optical properties.

On the one hand, in the case of TiO_2 and Ti-O-Si films, the pulse frequency was fixed at 1 kHz and the duty cycle was varied from 100 to 10%. Pulsing the power allows to decrease the deposition temperature (down to about 50°C) while conserving the anatase structure in the case of TiO_2 films, so that anatase and amorphous high refractive index Ti-Si-O films can be deposited on polymer substrates.

On the other hand, TTIP and HMDSO flow rates have been pulsed, either to get benefit from oxygen plasma treatment following oxide deposition or to deposit TiO_2/SiO_2 stacks.

[1] D. Li et al, Plasma Processes and Polymers, 2016

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2:40pm TF+PS+SE-MoA4 Plasma CVD of Boron-Carbon Thin Films from Organoboron Precursors for Next Generation Neutron Detectors, *Mewlude(Maiwulidan) Imam (Yimamu)*, Linköping University, Sweden; *C. Höglund*, Linköping University and European Spallation Source ERIC, Sweden; *R. Hall-Wilton*, European Spallation Source ERIC, Sweden; *J. Jensen*, Linköping University, Sweden; *S. Schmidt*, Linköping University and European Spallation Source ERIC, Sweden; *I.G. Ivanov, J. Birch, H. Pedersen*, Linköping University, Sweden

A novel design for neutron detectors based on thin films that are rich in the ¹⁰B isotope has been suggested for the European Spallation Source (ESS), in order to overcome the very limited availability of ³He. The detector design uses ¹⁰B₄C films deposited onto both sides of neutron transparent substrates, e.g., Al blades [1]. The use of aluminum (melting point at 660 °C) limits the deposition temperature for CVD processes and the use of chlorinated precursors due to etching of Al by HCl. Therefore, reactive organoborons are evaluated as precursors for these films using both thermal CVD [2, 3] and plasma CVD.

Plasma CVD of $B_x C$ thin films has been studied by introducing the organoborons trimethylboron B(CH₃)₃ (TMB) or triethylboron B(C₂H₅)₃ (TEB) into a microwave-induced Ar plasma without using any intentional substrate heating. The effect of plasma power, TMB or TEB to Ar ratio and total pressure on the film composition, morphology, density, chemical structure and internal stress were investigated by means of Tof-ERDA, SEM, XRR, XPS and HRXRD, respectively. Tof-ERDA results showed that the highest B/C ratio of 2 was achieved when using TMB at high plasma power. Densification of the films was accompanied by decreasing the total pressure below 0.4 mbar, resulting in a columnar film with densities of 2.16 \pm 0.01 g/cm³. The H content in the films was high (15 \pm 5 at. %) due to the low substrate temperature (~300 °C). XPS revealed that films deposited using TMB mainly contained B-C bonds and small contribution from C-C/CH bonds, that was evidenced by the observed amorphous carbon phases in the films by Raman spectroscopy. The internal compressive stresses in the films were increased with the Ar gas flow causing film delamination, while a low flow of Ar showed good adhesion and stress level is less than 300 MPa. In addition, the plasma composition studied by optical emission spectroscopy (OES) showed that BH, CH, C₂ and H lines were the most intensive lines in the spectrum. Considering the high H content in the films, we propose that BH and CH are the most likely species to contribute to the film formation.

- [1] R. Hall-Wilton et al. IEEE NSS/MIC conference record, 2012, 4283
- [2] H. Pedersen et al. Chem. Vap. Deposition2012, 18, 221
- [3] M. Imam et al. J. Mater. Chem. C2015, 3, 10898

3:00pm TF+PS+SE-MoA5 Plasma Enhanced Atomic Layer Deposition of Superconducting Nb_xTi_yN Films, *Mark Sowa*, Ultratech/CNT; Y. Yemane, J. Provine, Stanford University; E.W. Deguns, Ultratech/CNT; F. Prinz, Stanford University

NbN, TiN, and their mixtures have been studied for their use in superconducting applications. These materials are commonly deposited via sputtering techniques, but a lack of thickness control limits this technique from applying thin, uniform films. Atomic Layer Deposition (ALD) has been widely recognized for its ability to coat substrates with uniform film thicknesses ranging from a few Ångstroms to 100's of nanometers. Plasma Enhanced ALD (PEALD) extends the capabilities of the ALD technique, improving the properties of certain films, particularly nitrides deposited at low temperatures. PEALD of NbTiN has previously demonstrated superconducting properties¹ and PEALD NbN has been previously reported with a critical temperature of 10.4K².

In this work, Nb_xTi_yN ($0 \le x, y \le 1$) has been deposited using PEALD in an Ultratech/CNT Fiji system at substrate temperatures between 100 and 300°C. Stoichiometry was controlled by adjusting the ratio of NbN:TiN cycles during the film deposition. Precursors utilized for this study were (tbutylimido) tris(diethylamido) niobium (TBTDEN) and tetrakis(dimethylamido) titanium (TDMAT). A mixture of N2 and H2 was used as the plasma gas for the NbN cycles while TiN was deposited with an N2 plasma. Films were analyzed for thickness and optical properties through spectroscopic ellipsometry. Room temperature resistivity was derived from four point probe measurements. Samples were analyzed with X-ray photoelectron spectroscopy to determine stoichiometry and impurity levels. Superconductivity characteristics of the films will also be presented.

A 20nm, 300°C deposition of NbN, which had a room temperature resistivity of $282\mu\Omega\text{-}cm$, was shown to have a critical temperature of 12.4K and a critical field greater than 12 Tesla.

[1] E. F. C. Driessen, et al., "Strongly Disordered TiN and NbTiN s-Wave Superconductors Probed by Microwave Electrodynamics," Phys. Rev. Lett. 109, 107003, 2012.

[2] M. Ziegler, et al., "Superconducting niobium nitride thin films deposited by metal organic plasma-enhanced atomic layer deposition," Supercond. Sci. Technol. 26 (2013) 025008.

3:20pm TF+PS+SE-MoA6 Mechanical Reliability of PECVD Barrier Films for Flexible Electronics, *Kyungjin Kim*, A. Singh, H. Luo, T. Zhu, O. Pierron, S. Graham, Georgia Institute of Technology

The development of PECVD and ALD barrier films have proven to be viable approaches to create barrier films for flexible electronic applications. While much research has focused on the water vapor transport properties of these films, the mechanical reliability during flexural deformation is critical to the performance and durability of these coatings. Overall, the use of the critical onset strain is limiting in trying to define the limits of performance since it ignores time-dependent processes that can occur during mechanical deformation. In this work, we investigate the time-dependent channel crack growth behavior of silicon nitride and ALD barrier films on polyethylene substrates in humid and dry air. The evaluation of the cracking process versus applied strain and load was measuring in-situ using optical and laser scanning confocal microscopy. The results show that crack growth can occur at strains that are much lower than the standard measured onset critical strains. The results of the work show that both polymer relaxation of the PET substrate as well as environmentally assisted crack growth occurs in the films, both in a time dependent manner. Tests in dry air versus tests in humid air show crack growth rates increasing from 100 nm/s to 10 um/s for an applied stress intensity factor of 1.6 MPa.m^0.5. In addition to the dramatic changes in crack growth rates with environmental conditions, larger crack densities were observed in humid environments. This suggests an easier initiation and growth of crack in humidity versus dry air. Overall, the results presented will show the strong link between environment, temperature, and the rate at which cracks grow in barrier films. Finally, the energetics of the crack growth process will be presented as a better metric than onset crack strain to evaluate the mechanical reliability of the barriers for a given application.

4:00pm TF+PS+SE-MoA8 Origin of Stress in Sputtered CdTe and ZnS Films: Influence of Sputter Ion Mass on Mechanical and Chemical Layer Properties, *Ségolène Liénard*, Univ. Grenoble Alpes, LTM CNRS, 38000 Grenoble, France; *D. Sam-Giao, A. Kerlain*, Sofradir, BP 21-38113, Veurey-Voroize, France; *F. Boulard*, *C. Vallée*, Univ. Grenoble Alpes, France

Physical vapor deposition is a mature, well understood and established technology in integrated circuit fabrication. CdTe and ZnS binary II-VI compounds materials are commonly used in photovoltaic solar cells or infrared optics. However, sputtering deposition of these materials still suffer from a lack of comprehensive study to optimize process integration.

Our study is focused on the influence of projectile ions mass on properties of sputtered deposition CdTe and ZnS films. We compare physico-chemical, mechanical and electrical properties of CdTe and ZnS films deposited with Ar and Xe ions as sputter gas. Ar and Xe concentration in these films are characterized by Time of Flight Secondary Ions Mass Spectrometry (TOF-SIMS). Dedicated implanted reference samples are used to quantify the absolute concentration. Layers microstructures are characterized by Scanning Tunneling Electron Microscopy (STEM) and dielectric constant by capacitance-voltage measurements. We use the curvature method based on the well known Stoney concept [1] to calculate film stress while density is estimated by differential weighing.

With Ar ion deposition process (low sputter on target mass ratio), we observe Ar and cavities inside the CdTe layer. The density as well as the dielectric constant are below bulk values. A good agreement is found between the cavity density and the effective dielectric constant determined by the Bruggeman model [2-3]. on the contrary, Xe ion target sputtering (high sputter on target mass ratio) leads to denser films, without Xe inside the layers, and close to theory density and dielectric constant values. We discuss these observations in terms of backscattered ions incorporation. Moreover, the effect of thermal annealing time on stress evolution is discussed in regards of Ar or Xe incorporation and outgasing.

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[1] G.G. Stoney, Proc. Soc. London, A82, 1909, 172

[2] Aspnes D. E., Thin Solid Films 89 (1982) 249.

[3] Othman M.T., PhD "Spectroscopic Ellipsometry Analysis of Nanoporous Low Dielectric Constant films Processed via Supercritical CO2 for Nextgeneration Microelectronic Devices"., University of Missouri-Columbia, 2007

4:20pm TF+PS+SE-MoA9 Synthesis and Characterisation of MoB_{2-X} and Mo-B-C Thin Films by Non-Reactive DC Magnetron Sputtering, *Paulius Malinovskis*, Uppsala University, Sweden; *J.P. Palisaitis*, Linkoping University, Sweden; *P.O.A. Persson*, Linköping University, Sweden; *E.L. Lewin*, *U.J. Jansson*, Uppsala University, Sweden

Transition metal diborides (MeB₂) with the AlB₂-type structure have many unique properties such as high hardness, high conductivity and oxidation resistance One of the most studied diboride compounds is TiB₂ but also other transition metals like Cr, Nb and Mo can form the simple AlB₂ type structure.

Non-reactive magnetron sputtering is an excellent technique to deposit thin films of different MeB_2 phases. It is well-known that phases with rather simple crystal structures are preferably formed in magnetron sputtering where the quenching rates of the incoming atoms are high. Such metastable MeB_2 films may be chemically more reactive in a tribocontact and form a lubricating tribofilm of metal oxides and layered BO_x . Some metal oxides such as MoO_3 and boric acid (forming in humid atmosphere from BO_x) have been predicted to exhibit low friction coefficients. Consequently, it is possible that metastable MeB_2 films with the AlB_2 -structure may exhibit excellent low friction properties. Another way to tailor metal diboride properties is to alloy it with third element, e.g. carbon.

In this study we have investigated the microstructure, mechanical and tribological properties of DC magnetron sputtered MoB_{2-x} and Mo-B-C thin films from Mo/B and graphite carbon target. The films were characterized with XRD, XPS, TEM, nanoindentation and tribological ball-on-disk method. All films exhibited the AlB2-type structure with substoichiometric MeB2-x grains surrounded by a tissue phase of a-B and a-BCx. The MoB2-x films were substoichiometric with respect to boron and exhibited a much higher hardness compared to bulk samples, which could be attributed to a hardening effect of the tissue phase. Friction measurements confirmed the hypothesis that a significant tribofilm formation is present on the metastable MoB_{2-x} films. However, a reduced friction coefficient could not be observed. Addition of carbon resulted in a change in the composition of the tissue phase. This caused a reduction in hardness and a reduction of the friction coefficient. General trends in the phase formation and properties of Mo-B-C films will be explained in detail and compared with other Me-B-C systems (Me= Ti, Cr, Nb).

4:40pm TF+PS+SE-MoA10 Molybdenum Back Contacts Deposited by High Power Impulse Magnetron Sputtering, D.A. Loch, Arutiun Ehiasarian, Sheffield Hallam University, UK

Molybdenum thin films used in chalcopyrite solar cells can influence the Na diffusion rates and the texture of the Cu(InGa)Se₂ absorber according to the microstructure and morphology. The lowest resistivity films are achieved at low working pressure and are accompanied by high residual stress and poor adhesion due to the resulting high energy of the deposited flux. High Power Impulse Magnetron Sputtering was employed to ionise the sputtered flux, achieve high adatom mobility at low energy and influence the growth of Mo back contacts. Pulse durations in the range 60 to 1000 μ s, sputtering voltages between 800 and 1500 V and deposition pressures of 2×10⁻³ mbar and 4×10⁻³ mbar resulted in ten-fold variations in the flux ratios of Mo^{1+}/Mo^0 Mo^{2+}/Mo^{1+} , Ar^{2+}/Ar^{1+} and Mo^{1+}/Ar^{1+} as determined by optical emission spectroscopy and time-resolved plasmasampling energy-resolved mass spectroscopy. The energy of metal and gas double and single-charged ions reduced with pulse duration and increased with voltage. The microstructure of the films varied from open columnar with faceted tops to fully dense as observed by secondary electron microscopy. The reflectivity of the films improved by 20% compared to industry-standard materials. The lowest resistivity was in the range of 12 $\mu\Omega\text{-cm}$ as observed by four-point probe measurements of 570 nm thick films. The correlation between resistivity, microstructure, crystallographic texture, stress and deposition flux characteristics is discussed.

5:00pm **TF+PS+SE-MoA11 Plasma Characterization of Al and Cu with HIPIMS**, Jason Hrebik, Kurt J. Lesker Company; *R. Bandorf*, *H. Gerdes*, *D. Spreemann*, Fraunhofer Institute for Surface Engineering and Thin Films IST, Germany

High power impulse magnetron sputtering (HIPIMS) is a well-known technique for tailoring the coating properties in comparison to DC. In many cases the thin films were developed in smaller scale R&D facilities and afterwards transferred to industrial scaled machines. But the source configuration, magnetic field, and overall mechanical layout differs for the larger sputtering plant, and therefore a direct upscaling of the process is quite difficult. Since often the thin film properties are correlating with the plasma properties, plasma characterization is very useful tool for determining the main important parameters for a process transfer.

This investigation is focused on the plasma characterization of Al and Cu on a small circular target (3 inch) and will give a short comparison to a rectangular target (10 inch by 15 inch). As plasma properties the ion density and the optical emission was measured. The measurements were carried out in a time resoled mode and can be correlated to target voltage and current.

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