

Spectroscopic Ellipsometry Focus Topic Room 202A - Session EL+AS+EM-MoM

Application of SE for the Characterization of Thin Films and Nanostructures

Moderators: Alain C. Diebold, SUNY Polytechnic Institute, Mathias Schubert, University of Nebraska-Lincoln

8:20am **EL+AS+EM-MoM1 Stealth Technology-based Terahertz Frequency-domain Ellipsometry**, *Vanya Darakchieva*, Linköping University, Sweden
INVITED

We present the newly designed Terahertz (THz) frequency-domain spectroscopy (FDS) ellipsometer at the Terahertz Material Analysis Center (THeMAC) at Linköping university and demonstrate its application to a variety of technologically important materials and heterostructures. We show that employing concepts used in stealth technology for the instrument geometry and scattering anti-static coating, and modulation of the backward wave oscillator (BWO) THz source allows for effective suppression of standing waves enabling accurate ellipsometry measurements with high spectral resolution (of the order of MHz). We further demonstrate an etalon-based method for frequency calibration in THz FDS ellipsometry. The instrument can incorporate various sample compartments, such as a superconducting magnet, in-situ gas cells or resonant sample cavities, for example. Reflection and transmission ellipsometry measurements over a wide range of angles of incidence for isotropic (Si) and anisotropic (sapphire) bulk samples are presented together with determination of the material dielectric constants. We further demonstrate results from cavity enhanced THz optical Hall effect experiments on an AlGaIn/GaN high electron mobility transistor structure (HEMT), determining the free charge carrier density, mobility and effective mass parameters of the 2D electron gas (2DEG) at room temperature. We show through in-situ experiments on epitaxial monolayer graphene exposed to different gases and humidities that THz FDS ellipsometry is capable of determining free charge carrier properties and following their changes upon variation of ambient conditions in atomically thin layers. Exciting perspectives of applying THz FDS ellipsometry for exploring low-energy excitation phenomena in condensed and soft matter, such as the vibrational, charge and spin transport properties of magnetic nanolaminates, polymers and hybrid structures for photovoltaics and organic electronics; and determination of THz optical constants and signatures of security and metamaterials are envisioned.

9:00am **EL+AS+EM-MoM3 Spectroscopic Ellipsometry and Finite Element Modeling based Optical Characterization of Highly Coherent Au-Si Slanted Columnar Periodic Nanostructures**, *Ufuk Kilic*, University of Nebraska-Lincoln; *A. Mock*, Linköping University, Sweden; *R. Feder*, Fraunhofer IMWS, Germany; *D. Sekora*, *M. Hilffiker*, *R. Korlacki*, *E. Schubert*, *C. Argypopoulos*, *M. Schubert*, University of Nebraska-Lincoln

An unprecedented and phenomenal control of anisotropic optical properties of a material is reported here by utilizing periodic arrangement of nanostructures. These artificially engineered structures exhibit distinct optical, mechanical, and magnetic properties when they are compared with their bulk counterparts which has recently gained a growing interest due to its potential applications in various optical and optoelectronic systems such as lenses, solar cells, photodetectors, and sensors [1-3]. In addition to the material choices (ie. elemental composition), the size and shape of these artificial structures also play a key role in tailoring the aforementioned inherent properties.

Unraveling the mechanisms that influence and control the optical properties of highly-porous, periodic, and three-dimensional arrangements of nanoplasmonic structures can offer new approaches for the development of next generation sensors. Glancing angle deposition and atomic layer deposition can be used to create periodic nanostructures with multiple constituent materials, so-called heterostructured metamaterials.[4] In this study, we employ a two-source (ie. Au and Si) electron-beam-evaporated, ultra-high-vacuum glancing angle deposition which allows for the fabrication of highly-ordered and spatially-coherent super-lattice type Au-Si slanted columnar heterostructured thin films. We perform a combinatorial spectroscopic generalized ellipsometry and finite-element method calculation analysis to determine anisotropic optical properties. We observe the occurrence of a strong locally enhanced dark quadrupole plasmonic resonance mode (bow-tie mode) in the vicinity of the gold junctions, with a tunable and geometry dependent frequency in

the near-infrared spectral range. In addition, inter-band transition-like modes are observed in the visible to ultra-violet spectral regions. We demonstrate that changes in the index of refraction due to the concentration variation of a chemical substance environment (gaseous or liquid) within a porous nanoplasmonic structure can be detected by transmitted intensity alterations down to 1 ppm sensitivity.

References

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- [3] Frölich, Andreas, and Martin Wegener. *Optical Materials Express* 1.5 (2011): 883-889.
- [4] Sekora, Derek, et al. *Applied Surface Science* 421 (2017): 783-787.

9:20am **EL+AS+EM-MoM4 Temperature Dependent Dielectric Function and Critical Point Comparison of bulk Ge and α -Sn on InSb**, *Rigo Carrasco*, *C. Emminger*, *N. Samarasingha*, *F. Abadizaman*, *S. Zollner*, New Mexico State University

Germanium is an indirect bandgap semiconductor with a bandgap of 1.55 μm at room temperature. Its band gap can be shifted to longer wavelengths and becomes direct by adding 5-20% Sn, which allows to detect efficiently in the IR range. Alloys of Ge and Sn are therefore of interest for photovoltaics, detectors and room temperature lasers (2-7 μm). Alpha-tin on the other hand, is a semimetal that, when under strain, has a very small band gap at the Gamma point of the Brillouin zone. We compare this direct band gap (E_0 peak) occurring in the infrared region of strained α -Sn on InSb to the absorption edge of Ge.

We investigate the temperature dependence of the complex dielectric function (DF) and interband critical points (CPs) of bulk Ge between 10 and 738 K using spectroscopic ellipsometry in the spectral range from 0.5 to 6.3 eV at a 70° angle of incidence [1]. The complex dielectric function at each temperature is fitted using a parametric oscillator model. Figure 1 shows that variations in temperature influence structures in the spectra of the DF. Furthermore, we analyze CPs in reciprocal space by studying Fourier coefficients as described in [2]. The peaks of the E_0 and $E_0+\Delta_0$ CPs are relatively narrow (Fig. 2) which makes the analysis of their broadenings difficult. A small excitonic peak is visible at the absorption edge E_0 , also shown in Fig. 2.

Spectroscopic ellipsometry measurements were also performed on several epitaxially grown α -Sn layers on InSb in the spectral range of 0.03 to 6.5 eV. Comparing the results of the pseudo-dielectric function of Sn to the one of Ge shows a remarkable difference of both spectra in the IR- region, as demonstrated in Fig. 3. While structures at higher energies, such as the E_1 and $E_1+\Delta_1$ CPs, are similar in shape and amplitude for both materials, the E_0 -peak in α -Sn is significantly larger than in Ge. Therefore, we believe that the E_0 peak in the spectrum of Sn is not due to excitons but can probably be explained by other parameters which influence the band structure, such as strain, composition, or free carrier concentration. The large peak between E_0 and E_1 is an interference fringe. We also compare the temperature dependence of the E_0 gap in Ge and alpha-tin.

This work was supported by the National Science Foundation (DMR-1505172) and by the Army Research Office (W911NF-14-1-0072). C. Emminger gratefully acknowledges support from the Marshallplan-Jubiläumsstiftung.

References

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9:40am **EL+AS+EM-MoM5 Elastomer Thin Films and Conducting Nanostructures for Soft Electronics and Dielectric Elastomer Transducers**, *Bert Müller*, *B. Osmani*, *T. Töpfer*, University of Basel, Switzerland

Nanometer-thin polymer films are essential components of low-voltage dielectric elastomer transducers and will, for example, play a vital role in future artificial muscles [E. Fattorini et al.: *Ann. Biomed. Eng.* 44 (2016) 1355]. Organic molecular beam deposition (MBD) is a versatile technique to prepare silicone films under well-defined conditions [F. M. Weiss et al.: *Mater. Design* 105 (2016) 106; T. Töpfer et al.: *APL Mater.* 4 (2016) 056101], but the achievable growth rates of about 1 μm per hour are too low for the fabrication of multi-layer devices. Therefore, we have developed electro-spraying as an alternative deposition method with one or two orders of magnitude faster rates [F. M. Weiss et al.: *Adv. Electron. Mater.* 2 (2016) 1500476; F. Weiss et al.: *Langmuir* 32 (2016) 3276]. For the

two approaches, spectroscopic ellipsometry (SE) has been employed for in situ monitoring the film's optical properties, the film thickness and the surface morphology during deposition and ultra-violet (UV) light irradiation. The derived quantities were verified by means of atomic force microscopy (AFM). Subsequent to the silicone deposition and the cross-linking by UV light curing, Au has been deposited using MBD and sputtering. This deposition process was also quantitatively characterized using SE and controlled by means of the plasmonic fingerprints of the metal nanostructures [T. Töpfer et al.: Adv. Electron. Mater. 3 (2017) 1700073]. The ex situ AFM measurements revealed well-known modulations characteristic for strained surface layers [B. Osmani et al.: Eur. J. Nanomed. 9 (2017) 69]. Recent nano-indentation tests have demonstrated that the Au-layers on the silicone near the critical stress regime hardly contribute to the overall elastic modulus and are, therefore, a sound basis for smart electrodes [B. Osmani et al.: Adv. Mater. Technol. 2 (2017) 1700105]. The nano-mechanical probing of the powered thin-film dielectric elastomer transducers evidenced the importance of the thickness homogeneity for such devices [B. Osmani et al.: Appl. Phys. Lett. 111, (2017) 093104]. The function of planar thin-film dielectric elastomer transducers can be precisely determined taking advantage of the cantilever bending approach [B. Osmani et al.: Rev. Sci. Instrum. 87 (2016) 053901]. Spectroscopic ellipsometry and advanced atomic force microscopy with nano-indentation capability enables us to thoroughly characterize the film morphology as well as the optical and local mechanical parameters of silicone and Au/silicone nanostructures.

10:00am **EL+AS+EM-MoM6 Spectroscopic Ellipsometry Investigation of Temperature Effects in Heated Self-organized 2D Arrays of Au Nanoparticles**, *Michele Magnozzi, M. Ferrera, M. Canepa*, Università di Genova, Italy; *F. Bisio*, CNR-SPIN, Italy

Metal nanoparticles (NPs) have the interesting property of behaving as efficient converters of EM radiation into heat. While this can occur via interband photoexcitation, the presence of a Localized Surface Plasmon Resonance provides an extra degree of freedom to tune and optimize the heating [1].

Assessing the temperature of plasmonic NPs during or immediately after illumination is not an easy task, and typically involves the use of models that necessarily have to simplify the complex temperature-dependent dielectric and thermodynamic response of nanosystems; for this reason, a measurement of the T-dependent optical behavior of the NPs at well-defined, externally controlled T would greatly contribute towards a better understanding of the thermoplasmonic properties of metal NPs.

Spectroscopic ellipsometry (SE), being a high-sensitive and non-destructive technique, is an ideal tool to investigate the optical response of NPs systems, provided that a proper model is used for data analysis.

We report a T-dependent investigation of the optical response of densely-packed 2D arrays of gold nanoparticles supported on an insulating nanopatterned substrate [2]. SE measurements were acquired in the 245-1450 nm spectral range, under high-vacuum conditions and in the 25-350 °C temperature interval [3]. Using a dedicated effective medium approximation developed for this kind of systems [2], we are able to reproduce the complex anisotropic optical response of this system employing morphological parameters deduced by *ex-post* AFM analysis; the temperature-dependent dielectric functions of Au, required as input in the model, was obtained in a dedicated SE measurement. The model yields a very good agreement with experimental data at relatively low T; however, though the appropriate T-dependent dielectric function of Au is systematically employed, the model is no longer able to reproduce the data obtained at the highest T. Indeed, a satisfactory agreement is attained introducing an effective correction to the Drude term of the dielectric function of Au, that keeps into account morphological effects affecting the NPs surface - such as softening or melting - that enhance the surface electron scattering rate. Our analysis thus shows that the T-dependent optical properties of metal NPs deviate from simplified expectations, and validate SE as valuable tool to study the complex, anisotropic properties of plasmonic NPs systems.

References

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- [3] M. Magnozzi, F. Bisio, M. Canepa, *Appl. Surf. Sci.*, 421:651-655, 2017

10:40am **EL+AS+EM-MoM8 Spectroscopic Ellipsometry of 2D WSe₂ Films**, *Baokun Song, H.G. Gu, M.S. Fang*, Huazhong University of Science & Technology, China; *Y.L. Hong, W.C. Ren*, Shenyang National Laboratory for Materials Science Institute of Metal Research Chinese Academy of Sciences, China; *X.G. Chen, S.Y. Liu*, Huazhong University of Science & Technology, China

Recently, two-dimensional (2D) WSe₂ has become a popular choice for nanoelectronic, optoelectronic, and valleytronic devices due to its layer-modulated bandgap, high mobility ($\sim 200\text{cm}^2\text{V}^{-1}\text{s}^{-1}$) and on-off ratio (10^8), and large spin-orbit coupling effect. The performance of those novel WSe₂-based devices strongly depends on the intrinsic optical properties of WSe₂, which exhibit an intriguing layer dependency. Therefore, the accurate and quantitative characterization of the layer-dependent optical properties of WSe₂ is essential to the optimal design of those related devices.

In this work, the dielectric function, bandgaps, and critical points (CPs) of WSe₂ ranging from monolayer to bulk have been comprehensively investigated and analyzed by spectroscopic ellipsometry over an ultra-broad band (0.73-6.42eV). The dielectric function of high-quality uniform WSe₂ specimens prepared by chemical vapor deposition were firstly obtained from the ellipsometric spectra. Then the bandgaps of the WSe₂ films were determined from their corresponding absorption coefficient spectra. We experimentally observed that the bandgaps of the WSe₂ films change from 1.63eV in monolayer to 1.21eV in bulk. Moreover, by using the CPs analysis, a series CPs (A-H) in the dielectric function spectra were precisely distinguished and many of them were rarely reported before. The positions of CPs (A-E) exhibit an obvious red shift when the layer number increases, while the CPs (F-H) exhibit a slight blue shift. The former phenomenon can be partly interpreted as the decaying geometrical confinement of excitons, while the underlying reasons for the latter merit further studies. These novel and advanced optical features will promote the fundamental understanding of the electronic structures and the development of WSe₂-based devices.

11:00am **EL+AS+EM-MoM9 Thermal Evolution Process of MaPbI₃ Film Based on Spectroscopic Ellipsometry**, *X.Q. Wang, X.Y. Shan, H. Siddique, Rucheng Dai, Z.P. Wang, Z.J. Ding, Z.M. Zhang*, University of Science and Technology of China

Thermal Evolution Process of MaPbI₃ Film Based on Spectroscopic Ellipsometry

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Abstract

During the last few years, the hybrid organic-inorganic methylammonium lead halide perovskite CH₃NH₃PbI₃ (MaPbI₃) has received great interest in the field of photovoltaics [1,2]. The relevant researches develop rapidly since the first realization of organic-inorganic hybrid solar cell, due to the excellent performance of MaPbI₃, such as high charge mobilities, suitable band gap and long carrier diffusion length. However the stability of MaPbI₃ has been a key issue hinder the practical application [3]. Here we present in-situ spectroscopic ellipsometry measurement to understand the nature of thermal degradation process of MaPbI₃. The dynamic evolution process of dielectric constants of the as-prepared MaPbI₃ film through heating is obtained by an effective medium approximation model fitting. The proportion of MaPbI₃ and PbI₂ is also obtained from the analysis of the ellipsometry data. The thickness of the film decrease in two-step, which is explained as the collapse of the PbI₂ frame. Our work provide the first in-situ detection of the optical properties through the degradation process of MaPbI₃ film, which can be consulted for further improving the stability of MaPbI₃.

References:

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2. J.Y. Jeng *et al*, CH₃NH₃PbI₃ Perovskite/Fullerene Planar-Heterojunction Hybrid Solar Cells, *Advanced Materials* 25, 3727-3732 (2013).
3. J.H. Noh *et al*, Chemical Management for Colorful, Efficient, and Stable Inorganic–Organic Hybrid Nanostructured Solar Cells, *Nano Lett.* 13, 1764-1769 (2013).

Monday Morning, October 22, 2018

11:20am **EL+AS+EM-MoM10 a-Si as a Protective Layer to Block the Oxidization of Al mirrors**, *Yhoshua Wug*, University of California at Los Angeles; *D.D. Allred, R.S. Turley*, Brigham Young University

Arguably, the best path to produce a truly broadband, e.g., an IR-optical-UV-EUV (extreme ultraviolet) mirror, for a future space observatory is an EUV multilayer mirror coated by a very thin bare aluminum layer. However, using a bare Al layer presents challenges that first must be overcome. Al oxidizes rapidly when contact with the atmosphere occurs. The customary solution is to cover the mirror with a protective evaporated fluoride layer. Unfortunately, these are opaque under ~ 110 nm, whereas, bare Al itself is highly reflective down to 85nm and could be used as a mirror to that wavelength if a barrier were not required. Once the mirror is in space far from the Earth, where there is no oxygen, Al would no longer need a barrier layer. Could a barrier be removed in space? Neither fluorides nor aluminum oxide can be removed once they are deposited without damaging the mirror's surface and destroying VUV reflectance. a-Si could be used as a protective layer that is potentially removable without roughening the Al surface. Dry hydrogen etching processes exist that could remove a silicon barrier as silane gas which would dissipate quickly in space. Such a process would use the Al layer as an etch stopping barrier in removing the a-Si protective layer. But is a-Si a suitable barrier for Al? We report our variable-angle spectroscopic ellipsometry studies of evaporated a-Si thin films on evaporated Al films. We discuss the conditions where a-Si can act as a protective layer to block aluminum oxidation.

11:40am **EL+AS+EM-MoM11 Terahertz to Mid-infrared Dielectric Response of Poly-methacrylates for Stereolithographic Single Layer Assembly**, *D.B. Fullager, Serang Park, Y. Li, J. Reese*, University of North Carolina at Charlotte; *E. Sharma, S. Lee*, Harris Corporation; *S. Schöche, C.M. Herzinger, J.A. Woollam Co. Inc; G.D. Boreman, T. Hofmann*, University of North Carolina at Charlotte

Producing THz optical components with arbitrary shapes using additive manufacturing is receiving considerable interest because it offers a rapid, low-cost avenue for THz imaging system development. In order to design such THz optical components appropriately, accurate knowledge of the complex dielectric function of the materials used for stereolithographic 3D fabrication, is crucial. In this presentation we report on the complex dielectric function of several poly-methacrylates which are frequently used for stereolithographic fabrication. Spectroscopic ellipsometry data sets from the THz to mid-infrared spectral range were obtained from isotropically cross-linked poly-methacrylate samples. The data sets were analyzed using stratified layer optical model calculations using parameterized model dielectric functions. While the infrared spectral range is dominated by several strong absorption features with Gaussian profiles, these materials are found to exhibit only weak absorption in the THz range. In conclusion we find that thin transmissive THz optics can be easily achieved using poly-methacrylate-based stereolithographic fabrication. Possible origins of the observed absorption in the THz spectral range are identified and pathways to reduce it are discussed.

Spectroscopic Ellipsometry Focus Topic Room 202A - Session EL+EM-MoA

Spectroscopic Ellipsometry: Novel Applications and Theoretical Approaches

Moderators: Vanya Darakchieva/Stefan Zollner, New Mexico State University,

1:20pm **EL+EM-MoA1 The Physics of Low Symmetry Metal Oxides with Special Attention to Phonons, Plasmons and Excitons and their Potential for Uses in Power Electronics and Quantum Technologies, Mathias Schubert**, University of Nebraska - Lincoln, Linköping University, Sweden, Leibniz Institute for Polymer Research, Dresden, Germany; *A. Mock, R. Korlacki, S. Knight*, University of Nebraska - Lincoln; *V. Darakchieva*, Linköping University, Sweden; *B. Monemar*, Linköping University, Sweden, Tokyo University of Agriculture and Tech., Japan; *H. Murakami, Y. Kumagai*, Tokyo University of Agriculture and Technology, Japan; *K. Goto*, Tokyo University of Agriculture and Technology, Tamura Corporation, Japan; *M. Higashiwaki*, National Institute of Information and Communications Technology, Japan

INVITED

We discuss analysis of the dielectric function tensor for monoclinic metal oxides. We derive the dispersions of transverse, longitudinal and plasmon coupled modes in gallium oxide [M. Schubert et al., Phys. Rev. B 93, 125209 (1-18) (2016); Editors' Suggestion], the Lyddane-Sachs-Teller relation for monoclinic and triclinic semiconductors [M. Schubert, Phys. Rev. Lett. 117, 215502 (2016)], the identification of transverse and longitudinal phonons in scintillator material cadmium tungstate [A. Mock, M. Schubert et al., Phys. Rev. B 95, 165202 (1-15) (2017)], the band-to-band transitions and excitons and their eigenvectors in gallia [A. Mock, M. Schubert et al., Phys. Rev. B 96, 245205 (1-12) (2017)], the effective electron mass tensor measurement using the optical Hall effect in gallium oxide [S. Knight, A. Mock, M. Schubert et al., Appl. Phys. Lett. 112, 012103 (2018); Editors' Pick], the temperature dependence of band-to-band transitions energies in gallium oxide [A. Mock, M. Schubert et al., Appl. Phys. Lett. 112, 041905 (2018)], and the dielectric and inverse dielectric tensor analysis method for transverse and longitudinal phonon mode dispersion characterization in high-power laser material yttrium silicon oxide [A. Mock, M. Schubert et al., Phys. Rev. B, 97 165203 (1-17) (2018)].

2:00pm **EL+EM-MoA3 Mueller Matrix Spectroscopic Ellipsometry Based Scatterometry of Nanowire Gate-All-Around (GAA) Transistor Structures, M. Korde, Alain C. Diebold**, SUNY Polytechnic Institute

One of the most difficult measurement challenges facing semiconductor research and development is determining the feature dimensions and shape for complicated 3D structures. GAA transistors are fabricated from fins etched from a Si/Si_{1-x}Ge_x/Si/Si_{1-x}Ge_x/Si.. multilayer. (1, 2) At one point in the fabrication process, the nanowires used in GAA transistors have the nanowire transistor structures suspended between the source and drain. Considering the close spacing of neighboring transistors, measuring the nanowires is a significant challenge. In this talk, we present simulations aimed at understanding the sensitivity to changes in feature shape and dimension for the structures used to fabricate GAA transistors. Simulations of the multi-layer fins shown a clear sensitivity to fin shape and Si layer thickness which is enhanced by the use of the full Mueller Matrix capability vs traditional spectroscopic ellipsometry.

1. Optical measurement of feature dimensions and shapes by scatterometry, A.C. Diebold, A. Antonelli, N. Keller, APL Mat., (2018), in press.
2. Muller matrix spectroscopic ellipsometry based scatterometry simulations of Si and Si/Si_xGe_{1-x}/Si/Si_xGe_{1-x}/Si fins for sub 7-nm node gate-all-around transistor metrology, S. Dey, N. Keller, M. Korde, and Alain C. Diebold, SPIE, Metrology, Inspection, and Process Control for Microlithography XXXII, SPIE Advanced Lithography, San Jose, Feb 25-Mar. 1, 2018. To be published in conference proceedings.

2:20pm **EL+EM-MoA4 Anomaly in the Optical Constants of Ni near the Curie Temperature, Farzin Abadizaman, S. Zollner**, New Mexico State University

Magnetized Ni demonstrates an anomaly in its optical constants near the Curie temperature ($T_c = 627$ K). Experiment shows that this anomaly does not depend on the morphology of the sample. To investigate this feature, we have carried out a series of ellipsometry measurements in the energy

range of 0.5 to 6.5 eV as a function of temperature from 80 to 800 K in 25 K steps. Furthermore, temperature dependent Mueller Matrix (MM) measurements have been performed on the magnetized Ni at a single energy of 1.97 eV as a series of four runs, up and down, between 300 and 800 K. The MM data of magnetized Ni reveal slight changes in the anisotropic portion of the MM while passing T_c . However, vast changes in the isotropic MM elements are found, indicating that the anomaly is not due to the induced anisotropy of the sample. The anomaly occurs only in the first run when the temperature passes T_c , which suggests that it stems from magnetization. On the other hand, magnetizing the sample again and repeating the experiment do not demonstrate any anomaly as passing T_c , which disproves our previous suggestion. In fact, the anomaly happens only once for each sample, regardless of its morphology.

No observable variation in the out-of-plane grain sizes were found in the XRD data before and after the temperature measurement. This and the fact that the anomaly occurs for a single crystalline sample as well as for a polycrystalline one indicate that it cannot be due to the grain growth. This turned our attention to the surface effects: Several experiments have been conducted to investigate how the surface of sample changes around T_c and how cleaning the surface affects the optical constants of Ni. The authors believe that the anomaly around T_c originates from the surface changes. Yet the question of why it occurs near T_c is still open. The same series of experiments have been performed on cobalt and the results are compared to Ni.

2:40pm **EL+EM-MoA5 Phonon Confinement and Excitonic Absorption in the Optical Properties of ZnO Films, Nuwanjula Samarasingha, S. Zollner**, New Mexico State University; *D. Pal, A. Mathur, A. Singh, R. Singh, S. Chattopadhyay*, Indian Institute of Technology Indore, India

Wide band gap materials like ZnO, which have drawn much attention for optoelectronic devices, have a large excitonic binding energy of 60 meV at room temperature. These excitons directly influence the dielectric function (ϵ) of ZnO. Hence investigation of excitonic absorption on the optical properties is very important. Wurtzite type ZnO shows three excitonic peaks in the ordinary dielectric function which are directly related to the electronic band structure. Due to the spin orbit and crystal field splitting the top valence band of ZnO is split into three sub bands. The corresponding free exciton transitions between these three valence bands and the lowest conduction band are denoted by A, C, and B. We also observe an exciton-phonon complex.

We explore the behavior of phonons and excitons in c-oriented ZnO thin films grown on Si (smaller band gap than ZnO) and SiO₂ (larger band gap than ZnO) using variable angle UV spectroscopic ellipsometry and FTIR ellipsometry. In order to characterize the structural properties of our ZnO films we performed X-ray diffraction (XRD), X-ray reflectivity (XRR), and atomic force microscopy (AFM) measurements.

According to the UV ellipsometry data the real and imaginary parts of ϵ in thin ZnO films on Si are much smaller than in bulk ZnO. We find that the excitonic enhancement decreases monotonically with decreasing film thickness. A similar behavior can be seen for ZnO films on SiO₂ as a function of thickness. The impact of this excitonic absorption on the ϵ was described by Tanguy [1]. We will fit our ellipsometric spectra by describing the dielectric function of ZnO using the Tanguy model. We will investigate the dependence of the excitonic Tanguy parameters on film thickness and substrate material.

Wurtzite type ZnO has 12 phonon branches, 9 optical and 3 acoustic modes. Among these 9 optical modes, only 1A₁ and 1E₁ polar phonon modes are IR active. According to the IR ellipsometry data these IR active phonon mode frequencies of ZnO films are consistent with bulk ZnO. We find a small redshift and increasing broadening with decreasing ZnO film thickness on a Si substrate. We will analyze the thickness dependence of the phonon oscillator strength of ZnO films on Si and SiO₂ substrates.

Reference:

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Supported by NSF (DMR - 1505172).

Monday Afternoon, October 22, 2018

3:00pm EL+EM-MoA6 High Aspect Ratio Etch Tilt Detection with Full 4x4 Mueller Matrix Spectroscopic Ellipsometry and Its Application to 3DNAND Channel Hole Etch Process and Chamber Monitoring, *P. Ong*, Micron Semiconductor Asia Pte. Ltd., Singapore; *Shilin Ng*, Nanometrics Incorporated; *G.B. Chu*, Micron Semiconductor Asia Pte. Ltd., Singapore; *P. Murphy*, Nanometrics Incorporated; *L.C. Liang*, *W. Fu*, Micron Semiconductor Asia Pte. Ltd., Singapore; *Y. Wen*, Nanometrics Incorporated; *L.W. Ho*, Micron Semiconductor Asia Pte. Ltd., Singapore

Full 4x4 Mueller Matrix Spectroscopic Ellipsometry (MMSE)^[1] is a widely-used technique for measuring cross-sectional profile, critical dimensions (CD) and material thicknesses of repeating structures created as part of microelectronic device manufacturing processes. In this paper, it will be shown that its application can be extended to measuring asymmetries in such structures with off-diagonal Mueller Matrix Elements^[2]. These asymmetries, such as tilt of etched holes, and lines or trenches, are typically caused by inhomogeneity in the etch plasma sheath at the wafer edge. This paper will focus on one of the most important use-cases: tilt of high aspect ratio (HAR) etched 3D-NAND channel holes. Full 4x4 MMSE can be used to provide fast, accurate, non-destructive measurements of the channel hole tilt, both in direction and magnitude.

Furthermore, in contrast to CD and thickness measurements which are typically done in metrology test keys, this tilt measurement is in-die and on-device. This allows us to characterize the tilt at all locations on the wafer edge, as well as the variation in tilt as the wafer edge is approached. In addition, we also show how the measurements can be used to monitor the condition of the etch chamber for equipment control and/or to trigger preventive chamber maintenance.

3:40pm EL+EM-MoA8 Ultra-High-Speed Spectroscopic Ellipsometry and its Applications, *Gai Chin*, ULVAC Inc., Japan

As a comprehensive manufacturer of metrology tools and deposition tools, ULVAC developed an innovative high-speed spectroscopic ellipsometer for some thin-film deposition applications, such as PVD, CVD, ALD and others.

This novel spectroscopic ellipsometry can measure the thickness and optical constants of thin films at a dramatically fast speed. Its data acquisition time is as short as 10ms. It does not require any active components for polarization-control, such as a rotating compensator or an electro-optical modulator. The Fourier analysis of channeled spectrum obtained from the spectrometer allows determining the four spectroscopic ellipsometry parameters of the samples simultaneously.

It created great opportunities for new applications of the spectroscopic ellipsometry in which the compactness, the simplicity and the rapid response are extremely important. It can be integrated into the deposition tool and successfully measured thin films in-situ to realize the Advanced Process Control (APC). Obviously, those from PVD, CVD and ALD are some promising applications for this novel spectroscopic ellipsometry.

This paper describes the principle, system configuration and creative efforts on developing a series of high-speed spectroscopic ellipsometers. Some of its new applications will be also introduced, such as the PVD, CVD, ALD, EUV, OLED, MEMS and some measurement data of thin films from the semiconductor, flat panel display and other industries.

4:00pm EL+EM-MoA9 Use of Ellipsometry to Monitor Implant Damage in Methane Plasma Implant, *Nicholas Bateman*, Varian Semiconductor Equipment, Applied Materials

The Applied Materials PLAD tool consists of an inductively couple plasma source and a pulsed direct current (DC) bias coupled to a platen upon which a wafer is e-chucked. This tool architecture enables high throughput for high dose implants. Unlike a standard beamline implant, which will not lead to any deposition on the wafer, the plasma implant process directly exposes the wafer to the plasma leading to both deposition and implant damage. Standard industrial metrologies like Thermawave [1] are sensitive only to the implant damage. Process control could be enhanced if both the implant damage and the deposition thickness could be monitored online.

Ellipsometry is extensively used in the semiconductor industry to measure and monitor film thickness and optical properties [2]. It would be the ideal industrial technique to measure the deposition left by a plasma implant process, but has not been used to evaluate the damage caused by ion implant.

This work presents the development of an ellipsometry model that can be used to simultaneously monitor the deposited layer thickness and the implant damage caused by a methane plasma implant. The dispersion function for the deposited layer was determined by fitting multi-angle, broad wavelength ellipsometry data for different process times and bias

conditions. The extracted thickness of the damage layer is shown to be well correlated to Thermawave across a wide range of process parameters, and through an extended 'marathon' test. As the implant voltage is reduced to zero the model trends continuously to match the results of a 'deposition only' ellipsometry model that matches SEM thickness measurements.

These results suggest that for plasma doping applications, ellipsometry can be used to monitor both implant damage and deposition simultaneously to allow improved process control.

References:

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4:20pm EL+EM-MoA10 Study of the Thickness-dependent Optical Constants of Metallic Thin Films based on Ellipsometry and Reflectivity, *Jiamin Liu*, *H. Jiang*, *S.Y. Liu*, Huazhong University of Science and Technology, China

Metallic thin films have been widely used in various plasmonic and nanophotonic applications, such as bio-chemical sensors, meta-materials and nanolasers, benefiting by their size-dependent optical constants which are different from that of bulk materials. Considering that the performances of these films are sensitive to their thicknesses and optical constants, it is highly desirable to precisely characterize the thicknesses and the optical constants of such thin films for better applications.

In this work, a synergic analysis method based on ellipsometric parameters and reflectivity has been proposed, which enables the simultaneous determination of both the thickness and the optical constants for the metallic thin film. Both the ellipsometric parameters, including the amplitude ratio $\tan(\psi)$ and the phase difference Δ between p- and s-components, as well as the reflectivity are acquired using one ellipsometer. The proposed method consists of a point-by-point synergic regression analysis on the reflectivity and the ellipsometric parameters as well as an oscillator-parametrization regression analysis on the ellipsometric parameters. The former analysis allows for the accurate determination of the thickness of metallic thin films, while the latter enables the acquisition of the optical constants. Both virtual and practical experiments of measuring a series of Cu thin films deposited on Si substrates have been sequentially carried out for demonstration. The results clearly show the coupling effect between the thickness and optical constants of these samples. And by comparing with the thicknesses reported by AFM and TEM, the validity and the accuracy of the proposed method have been verified. Further analysis on the optical constants of Cu thin films has been carried out using the oscillator-parametrization we proposed, in which the thickness dependency of the Drude term, the plasma energy and the relaxation time has also been analyzed.

Tuesday Evening Poster Sessions, October 23, 2018

Spectroscopic Ellipsometry Focus Topic

Room Hall B - Session EL-TuP

Spectroscopic Ellipsometry Focus Topic Poster Session

Moderator: Tino Hofmann, University of North Carolina at Charlotte

EL-TuP1 An In situ Spectroscopic Ellipsometry Study of Cerium Oxidation, *Wayne Lake, P. Roussel, AWE, UK*

Cerium is an electropositive metal and will be covered by an oxide film. X-ray Photoelectron Spectroscopy (XPS) measurements have shown the oxide film to be composed of the trivalent oxide at the metal interface and the tetravalent dioxide at the oxide gas interface. Furthermore, in ultra high vacuum the dioxide film is thermodynamically unstable with respect to the cerium metal substrate and reduces to the trivalent oxide. The XPS technique is limited due to the small depth probed, therefore, to follow cerium oxidation reaction with oxide films greater than 10 nm spectroscopic ellipsometry offers a better technique of choice. At the AWE the spectroscopic ellipsometer is attached to an in situ film growth chamber on the XPS spectrometer. The problem with spectroscopic ellipsometry arises from the data interpretation. Spectroscopic ellipsometry modelling of a substrate with a single oxide film is easily achieved. However, when two oxides of different oxidation state are formed this presents a more difficult challenge to model.

Starting from sputter cleaned cerium substrate, the sample is heated and exposed to oxygen and the reaction is followed by using in situ spectroscopic ellipsometry. The substrate model is determined from the first data points in the data set prior to exposing the sample to oxygen. This work addresses how we determine a suitable model to interpret the spectroscopic ellipsometry data where two oxides are present.

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EL-TuP2 In-situ Multi-wavelength Ellipsometric Monitoring of the Reactive Sputter Deposition of WO_x Films, *Ned Ianno, G. Kaufman, C. Luth, University of Nebraska-Lincoln; C. Exstrom, S.A. Darveau, University of Nebraska at Kearney; B. Johs, Film Sense*

Thin films of WO_x where $x < 3$ have a range of applications as sensors, while amorphous WO_3 thin films have been employed as the precursor films for the growth of WSe_2 and WS_2 films. The WO_x films have been deposited by reactive sputtering in an Oxygen/Ar ambient, while the WO_3 films have primarily deposited by thermal evaporation although reactive sputter deposition has also been reported. Based on the literature and the work reported here the reactive sputter deposition of WO_x is very dependent on plasma bombardment during growth, and the voltage applied to the sputter gun, as well as the more straightforward parameters such as pressure, flow rate and substrate temperature. In view of this we have performed a 2-level factorial survey of the deposition parameter space associated with the reactive sputter deposition of WO_x films where we varied the O/Ar flow rate ratio, total chamber pressure, substrate temperature and sputter gun magnetic configuration to provide a more fundamental understanding of the deposition of WO_x films. A critical part of this work is the use of in-situ multi-wavelength ellipsometry (data acquired at 4 wavelengths in the visible spectrum: blue, green, yellow, and red) to monitor the growth process where we will show the sensitivity of the ellipsometric data to stoichiometry of the film, both during deposition and post deposition annealing.

EL-TuP3 Mid-infrared Optical Constants of InAsSb Alloys and Bulk GaSb, *Pablo Paradis, S. Zollner, R. Carrasco, New Mexico State University, Department of Physics; J. Carlin, V. Dahiya, A. Kazemi, S. Krishna, The Ohio State University, Department of Electrical and Computer Engineering*

Antimonides are attractive materials for mid-infrared detectors and emitters, because they form a direct band gap, which can be tuned from 0.1 to 0.7 eV. For the design and modeling of such devices, the optical constants of these materials must be known. We present results of Fourier-transform infrared (FTIR) ellipsometry measurements of bulk GaSb and doped and undoped InAsSb alloys with different compositions. Doped and undoped layers of InAsSb alloys were grown on GaSb substrates by MOCVD. Their optical constants were determined using two different methods. First, we fitted the data as a sum of oscillators representing the free-carrier and interband optical response, which allows a physical interpretation of the results. Second, we expanded the dielectric function into a sum of Kramers-Kronig consistent B-spline polynomials, assuming thicknesses obtained from the growth parameters. In the doped layers, a

free-carrier reflectance band can clearly be seen in the spectra, while the undoped layers show an absorption increase at the band gap. The 10% Sb samples are lattice matched and the 44% Sb samples are lattice mismatched leading to some strain inhomogeneity in the samples. This can be seen in the dielectric function of these samples. In the doped samples, we analyze the optical conductivity obtained from parametric oscillator fit. We pay attention to the plasma frequency term in our parameters to describe the behavior of the conductivity in doped vs undoped layers.

EL-TuP4 Temperature-dependent Ellipsometry and Thermal Stability of $Ge_2Sb_2Te_5$:C Phase Change Memory Alloys, *Cesy Zamarripa, N. Samarasingha, F. Abadizaman, R. Carrasco, S. Zollner, New Mexico State University*

$Ge_2Sb_2Te_5$ (GST) compounds are phase change memory alloys. At temperatures above 425 K, they are crystalline, forming a metastable rocksalt ($T > 425$ K) or a stable hexagonal crystal structure ($T > 525$ K). Heating the alloys above their melting point, followed by rapid cooling to room temperature (on a nanosecond time scale) forms an amorphous phase, where the resistivity is at least three orders of magnitude higher than in the crystalline phase.¹ This enables their use as rewritable optical recording media. Carbon doping allows tuning of the amorphous to crystalline transition temperature. In this work, we performed temperature-dependent spectroscopic ellipsometry measurements of as-deposited (amorphous) GST alloys in high vacuum from 300 to 800 K in 25 K steps, at an incidence angle of 70°. The samples were held approximately three hours at each temperature. We used two different instruments, a J.A. Woollam Fourier-transform infrared ellipsometer with ZnSe windows from 0.07 to 0.7 eV and a J.A. Woollam VASE ellipsometer with quartz windows from 0.5 to 6 eV. The GST layers were about 750 nm thick and deposited on singlet side polished Si wafers covered with 400 nm of SiO_2 . The original room-temperature measurements show two sets of interference fringes below 1 eV, due to the two different films present on the wafer. The SiO_2 absorption bands at 0.15 eV are clearly visible. The GST layers are transparent in the infrared without any lattice vibration features, due to the large mass of the constituent atoms. The absorption rises rapidly at 1 eV towards a broad maximum at 1.7 eV and then drops smoothly towards the UV. The dielectric function of the as-deposited films is featureless, as expected for an amorphous layer. The ellipsometric spectra are essentially unchanged between 300 and 400 K, showing an absorption threshold near 1.1 eV. At 425 K, this threshold suddenly drops to 0.7-0.8 eV, where it remains constant up to 675 K. The dielectric function is featureless at all energies and never displays any sharp features expected for a crystalline material. No amorphous to crystalline phase transition can be observed in the optical spectra. Spectra above 700 K show only the interference oscillations from the SiO_2 oxide layer. Apparently, the GST film has evaporated.

Acknowledgments: CMZ acknowledges support from the New Mexico Alliance for Minority Participation. This work was supported by NSF (DMR-1505172).

1. E.M. Vinood, K. Ramesh, and K.S. Sangunni, Sci. Rep. 5, 8050 (2015).

Plasma Science and Technology Division Room 104B - Session PS+AS+EL+EM+SE-WeM

Current and Future Stars of the AVS Symposium I

Moderator: Eric A. Joseph, IBM Research Division, T.J. Watson Research Center

8:20am **PS+AS+EL+EM+SE-WeM2 Invited Talk-Future Stars of AVS Session: Ellipsometry at THz Frequencies: New Approaches for Metrology and Metamaterial-based Sensing, Tino Hofmann¹**, University of North Carolina at Charlotte

Spectroscopic ellipsometry at terahertz frequencies has seen substantial advancements over the last several years. Now, instruments are available which allow precise measurements of the material's complex dielectric function including its anisotropy. This access to accurate electromagnetic material properties at THz frequencies is essential for the development of increasingly advanced THz optical systems and a prerequisite for the design and manufacturing of optical elements for this spectral range.

In this talk I will give an overview of recent developments in the implementation of THz ellipsometry and focus on applications where THz ellipsometry contributed valuable material parameters. In combination with external magnetic fields generalized THz ellipsometry allows the accurate measurement of the optical Hall effect. The optical Hall effect enables the precise determination of the free charge carrier properties effective mass, mobility, and density in semiconductor heterostructures at THz frequencies without the need of electrical contacts and will be discussed in detail.

The exploration of novel physical phenomena observed in artificially structured metamaterials and the application thereof is of interest due to its relevance for the design and fabrication of novel THz optical elements and sensors. Metamaterials have attracted continued interest for almost two decades due to their unique electromagnetic properties, which can differ substantially from their constituents and often do not even exist in naturally occurring materials. We have demonstrated that although being orders of magnitude smaller than the probing wavelength, metamaterials composed of highly-ordered 3-dimensional metal nanostructures exhibit a strong anisotropic optical response at THz frequencies. I will discuss how these interesting optical properties may be used for novel THz sensor and device designs.

8:40am **PS+AS+EL+EM+SE-WeM3 Invited Talk-Future Stars of AVS Session: Remote Epitaxy – The Future for Stackable SiC Electronics, Rachael Myers-Ward²**, U.S. Naval Research Laboratory; *J. Kim*, Massachusetts Institute of Technology; *M.T. DeJarlid*, US Naval Research Laboratory; *K. Qiao*, *Y. Kim*, Massachusetts Institute of Technology; *S.P. Pavunny*, *D.K. Gaskill*, U.S. Naval Research Laboratory

Ideally, electronic heterostructures from dissimilar materials leads to enhanced functionality. Yet, experimentally forming these heterostructures is challenging due to lattice or thermal coefficient of expansion mismatch leading to defect formation or thermally driven atomic diffusion resulting in cross-doping and gradual junction transitions. These challenges may be overcome with the discovery of remote epitaxy and 2D layer transfer [1]. Here, SiC epitaxy is performed on epitaxial graphene as the electrostatic fields from the substrate penetrate the graphene and guide adatom registry. The film is easily peeled away since the graphene is not bonded to either the substrate or epilayer; the epilayer is then van der Waals bonded to a different material enabling new functionality. We will present experimental results on the remote epitaxy of SiC, illustrating potential quantum science applications.

There are three necessary steps to create remote epitaxy. The first is to grow epitaxial graphene on SiC, followed by transferring the graphene to a desired substrate (if different from SiC), and finally the growth of the remote epitaxial layer. If the remote epitaxy is to be SiC, which is the focus of this paper, the second step is not needed. Epitaxial graphene (EG) was first synthesized on 4H- and 6H-SiC in a horizontal hot-wall CVD reactor between 1540 and 1580 °C in 10 slm of Ar and 100 mbar [2]. The growth temperature was dependent upon the offset of the substrate, where substrates with higher offsets require a lower growth temperature to ensure 1 ML of EG, which is desired to assist in SiC adatom registry during growth. SiC remote epitaxy was then performed on the EG using silane (2%

in H₂) and propane precursors, where the SiC polytype replicated the underlying substrate. In an effort to transfer the remote SiC epi/EG to another substrate such as SiO₂/Si, a metallization step was performed. Thin Ti and/or Ni layers were initially deposited followed by a thicker high stress metal to create strain and aid in removing the remote SiC epi/EG from the SiC substrate [1]. Once transferred, the metal was removed via a metal etch.

In this work, we will discuss the important parameters needed for successful remote SiC epitaxy, such as metallization, graphene thickness and remote epitaxy growth temperature. The epitaxial morphology characterized by SEM and Nomarski microscopy and graphene coverage and transfer evaluated by Raman spectroscopy will be presented.

[1] Kim, *et al.*, Nature 544, 340 (2017).

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9:00am **PS+AS+EL+EM+SE-WeM4 Invited Talk-Future Stars of AVS Session: Low-Temperature Growth for 3D Integration of van der Waals Materials, Christopher L. Hinkle³**, University of Texas at Dallas

The integration of novel logic and memory devices, fabricated from van der Waals materials, into CMOS process flows with a goal of improving system-level Energy-Delay-Product (EDP) for data abundant applications will be discussed. Focusing on materials growth and integration techniques that utilize non-equilibrium, kinetically restricted strategies, coupled with in-situ characterization, enables the realization of atomic configurations and materials that are challenging to make but once attained, display enhanced and unique properties. These strategies become necessary for most future technologies where thermal budgets are constrained and conformal growth over selective areas and 3-dimensional structures are required.

In this work, we demonstrate the high-quality MBE heterostructure growth of various layered materials by van der Waals epitaxy (VDWE). The coupling of different types of van der Waals materials including transition metal dichalcogenide thin films (e.g., WSe₂, WTe₂, HfSe₂), helical Te thin films, and topological insulators (e.g., Bi₂Se₃) allows for the fabrication of novel electronic devices that take advantage of unique quantum confinement and spin-based characteristics. We demonstrate how the van der Waals interactions allow for heteroepitaxy of significantly lattice-mismatched materials without strain or misfit dislocations. We will discuss TMDs, Te, and TIs grown on atomic layer deposited (ALD) high-k oxides on a Si platform as well as flexible substrates and demonstrate field-effect transistors with back-end-of-line (<450 °C) and even flexible plastics (<200 °C) compatible fabrication temperatures. High performance transistors with field-effect mobilities as high as 700 cm²/V-s are demonstrated. The achievement of high-mobility transistor channels at low processing temperatures shows the potential for integrating van der Waals materials into new technologies.

This work was supported in part by the Center for Low Energy Systems Technology (LEAST), one of six centers supported by the STARnet phase of the Focus Center Research Program (FCRP), a Semiconductor Research Corporation program sponsored by MARCO and DARPA. This work is also supported in part by NEWLIMITS, a center in nCORE, a Semiconductor Research Corporation (SRC) program sponsored by NIST through award number 70NANB17H041.

9:20am **PS+AS+EL+EM+SE-WeM5 Invited Talk-Future Stars of AVS Session: Engineering the Properties at Heusler Interfaces, Jason Kawasaki⁴**, University of Wisconsin - Madison

The Heusler compounds are a ripe platform for engineering and discovering emergent electronic, magnetic, topological, and ferroic properties at crystalline interfaces, either with other functional Heuslers or with compound semiconductor or oxide substrates. In these applications, the ability to control interfaces with near atomic level control is of tantamount importance; however, challenges such as interdiffusion have hampered their development. Here, I will discuss our efforts to control the properties of Heusler interfaces using precision growth by molecular beam epitaxy (MBE). Results will be presented in three areas: (1) the use of epitaxial strain to stabilize the hexagonal phase of several polar metal candidates, (2) the use of monolayer graphene diffusion barriers to enable high temperature growth and performance of spintronic devices, and (3) the phase segregation of ferromagnetic FeV nanostructures from a semiconducting FeVsb matrix with coherent epitaxial interfaces. Together, these examples illustrate the power of epitaxy and interfaces in controlling

¹ Future Stars of the AVS

² Future Stars of the AVS

Wednesday Morning, October 24, 2018

³ Future Stars of the AVS

⁴ Future Stars of the AVS

Wednesday Morning, October 24, 2018

the properties of Heuslers and other intermetallic compounds, and integrating them onto commonly used semiconductor substrate platforms.

9:40am **PS+AS+EL+EM+SE-WeM6 Invited Talk-Future Star of AVS Session: Atom Probe Tomography for 3D Semiconductor Devices Applications, Ajay Kumar Kambham²**, GLOBALFOUNDRIES U.S. Inc.

Device structures are rapidly scaling down to the nanometer regime with the ongoing development in semiconductor device technology. Along with this, it is ever critical need to engineer dopant profiles and to define the formation of junctions in Metal-oxide field effect transistors (MOSFETs). This is increasingly challenging considering the severity of short channel effects (SCEs). Indeed, one type of SCE in MOSFET devices known to cause performance degradation is Drain Induced Barrier Lowering (DIBL). To reduce DIBL, dopant junction profiles are made more abrupt. This can be done through the introduction of Sigma/cavity, fully depleted silicon-on-insulator (FDSOI) structures and the modulation of stress through optimal engineered epitaxial buffer layers. To assess the quality over nanometer scale regions requires the use of analysis techniques such as Atom Probe Tomography (APT) and Transmission Electron Microscopy (TEM). This presentation will discuss the role of APT and how elemental distributions vary depending on process conditions along with the challenges involved in sample preparation.

11:00am **PS+AS+EL+EM+SE-WeM10 Invited Talk-Future Stars of AVS Session: Three-Dimensional Imaging of Complex Oxide Interfaces, Divine P. Kumah²**, North Carolina State University

Complex oxide materials have a wide range of exciting tunable electronic and magnetic phases including ferroelectricity and superconductivity. The ability to fabricate atomic layers of complex oxides has led to the formation of novel interfaces and heterostructures of scientific and technological interest. The functional properties are usually correlated to sub-Angstrom structural perturbations at these interfaces. In this talk, a non-destructive synchrotron X-ray three-dimensional imaging technique will be applied to understand thickness-dependent electronic and magnetic transitions which occur in rare-earth manganite films with thicknesses on the order of an atomic layer. We show that structural distortions arising due to the electrostatic interfacial boundary conditions of the thin films are related to their thickness-dependent phase transitions. Based on these results, we show that heterostructures can be designed by molecular beam epitaxy to tune the atomic-scale structure of the manganite films to achieve robust ferromagnetism in atomically-thin layers. These results have important implications for the design of oxide-based spintronic devices and provide an important pathway for the realization of novel functional materials.

11:20am **PS+AS+EL+EM+SE-WeM11 Invited Talk-Future Stars of AVS Session: Illuminating Physics of Magnetron Sputtering Discharges, Matjaz Panjan³**, Jozef Stefan Institute, Slovenia

Magnetron sputtering is an established plasma technology for the deposition of thin films. In general, the technique is classified by the voltage supplied to the cathode; this can be continuous (DCMS), pulsed (HiPIMS) or oscillatory (RFMS). The distinction is also made with respect to the geometry of the magnetron source (e.g., circular, linear, cylindrical) and the magnetic field configuration (balanced or unbalanced). Despite the differences in the cathode operation, geometry and, magnetic field configuration, the underlying principle that forms dense magnetron plasma is the same. The central feature of magnetron sources is a crossed magnetic and electric field arrangement, which captures electrons close to the cathode. In such configuration, electrons gyrate around the magnetic field lines, bounce from the electric field of the sheath and drift in the azimuthal direction. The entrapment of electrons increases the plasma density close to the cathode (e.g., forming a ring-shaped plasma above the circular magnetron) and enhances the sputtering rate. Experiments using high-speed imaging and other techniques revealed that magnetron plasma is not azimuthally homogenous instead, it forms dense plasma structures called spokes. These structures have been extensively studied over the past few years and have changed our understanding of several physical processes in the magnetron discharges.

Spokes are observed for a wide range of discharge conditions, magnetron geometries and are an essential feature of all operational regimes [1-3]. They commonly form periodic patterns, have an arrowhead-like shape with an arrow pointing in the $\mathbf{E} \times \mathbf{B}$ direction, and travel with azimuthal velocities of several km/s. In the talk, I will present efforts to understand the physics

of spokes and magnetron discharges in general. In particular, I will discuss spatial distribution of the plasma potential [4] and the influence it has on the transport of charged particles [5], sputtering process and overall sustainability of the discharge. I will demonstrate that electric fields associated with spokes cause localized re-energization of electrons and thus help to sustain magnetron discharge. Spokes also influence energy and spatial distribution of ions and therefore indirectly affect the thin film growth.

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[3] M. Panjan *et al.*, *Plasma Sources Sci. Technol.*, **24** (2015) 065010

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11:40am **PS+AS+EL+EM+SE-WeM12 Peter Mark Memorial Award: Plasma-bio Interactions: Investigating Mechanisms to Enable New Applications, Peter Bruggeman⁴**, University of Minnesota

INVITED

Cold non-equilibrium atmospheric pressure plasmas (CAPs) have received a lot of attention in the last decades due to their huge potential for biomedical applications including wound healing, cancer treatment, dental treatments and disinfection and decontamination of heat sensitive materials [1]. These applications are due to the near ambient gas temperature at which CAPs can be produced and their high reactivity, involving the production of numerous reactive oxygen and nitrogen species [2]. Many applications require controlled interactions of plasma with bacteria, virus and mammalian cells or tissue that enable selectivity between healthy and cancer cells or in the treatment of bacteria on healthy tissue or food samples for which off target effects needs to be minimized. A controlled selectivity might be the greatest challenge for these applications and requires a detailed understanding of the underlying plasma-bio-interaction mechanisms. In this framework, my group in collaboration with microbiologists has performed detailed studies of the interactions of CAP with virus, bacteria and mammalian cells. Our research shows that controlling the gas phase plasma chemistry can lead to significant different biological responses of the living organisms [3-6]. The outcomes of these studies allow unraveling chemical pathways responsible for plasma-bio interactions and linking plasma kinetics to plasma-bio interactions. These insights are of invaluable importance for the development of applications in the field of plasma medicine.

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Acknowledgements

This work is partially supported by the "Plasma Science Center on Control of Plasma Kinetics" of the United States Department of Energy, Office of Fusion Energy Science (DE-SC0001319), the Agriculture and Food Research Initiative of the USDA's National Institute of Food and Agriculture (2017-67017-26172) and a Department of Energy Early Career Research Award (DE-SC0016053).

¹ Future Stars of the AVS

² Future Stars of the AVS

³ Future Stars of the AVS

Thin Films Division

Room 102A - Session TF+AS+EL+PS-ThM

In-situ Characterization and Modeling of Thin Film Processes

Moderator: Thomas Riedl, University of Wuppertal

8:00am **TF+AS+EL+PS-ThM1 Defects in Thin Films: A First Principles Perspective**, *Douglas Irving, J.S. Harris, J.N. Baker, S. Washiyama, M.H. Breckenridge*, North Carolina State University; *P. Reddy*, Adroit Materials;

R. Collazo, Z. Sitar, North Carolina State University **INVITED**

Realization of next-generation power and optoelectronic devices depends on the ability to controllably donor dope thin films of AlN and Al-rich AlGaN. The challenge in donor doping these materials begins with the donor dopant itself, Silicon. While it is a common shallow donor dopant in GaN, it exhibits a deeper ionization level in AlN due to the formation of a DX center near the conduction band minimum. Compensation in both the low and the high doping regime also presents a significant technical challenge to the doping of AlN thin films. In this talk, we explore the mechanisms for compensation in Si-doped AlN in the low and high doping regimes. For this purpose, we have implemented first principles density functional theory calculations with screened hybrid exchange-correlation functionals to determine the properties of individual defects in AlN. The formation energies of each defect are used within a grand canonical equilibrium model to identify the predominant defects as a function of growth conditions. In the low doping regime, important to drift layers in power electronics, we find unintentional impurities and unintentional impurity complexes are often responsible for free carrier compensation. Compensation in films that are doped to higher impurity concentration is found to be related to vacancy-dopant complexes. Possible solutions unique to thin films have also been explored and will be presented. Results from these methods are compared with complementary experimental data that includes below band gap optical absorption and photoluminescence, electrical measurements, dopant implantation, and available SIMS measurements.

8:40am **TF+AS+EL+PS-ThM3 Advances in Numerical Simulation of SiN ALD**, *Paul Moroz*, TEL Technology Center, America, LLC

Atomic layer deposition (ALD) includes a fast growing area of applications and could be foreseen as becoming one of the leading semiconductor technologies. In many cases, it allows accurate atomic-scale deposition of films with almost conformal profiles. Here we present new results on the Monte Carlo feature-scale simulations of ALD conducted with a feature-profile simulator, FPS3D [1-5], as well as comparison of obtained simulation results with the corresponding experiments. The ALD processes are often complex, involving large molecules and, to our knowledge, have not been addressed by other feature-profile simulations except FPS3D. The main factor of all of ALD schemes is the cyclic change in flux parameters and in the corresponding surface chemistry, which results in a single monolayer or, most typically, in a fraction of a monolayer of a film deposited after application of a cycle. Here, we consider a case of ALD with two time-steps: (1) dichlorosilane gas and (2) ammonia plasma. The SiN deposition rate in this case is about a half of a monolayer per cycle. A set of surface reactions is considered which emphasize the steric hindrance effect that was found to be an important factor in explaining deposition rates for this ALD process.

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9:00am **TF+AS+EL+PS-ThM4 Diffusion Kinetics Study of Adatom Islands: Activation Energy Barriers Predicted using Data-driven Approaches**, *ShreeRam Acharya, T.S. Rahman*, University of Central Florida

The Self-Learning Kinetic Monte Carlo (SLKMC) method [1] with a pattern recognition [2] and a diffusion path finder scheme enables collection of a large database of diffusion processes including single- and multiple-atoms, and concerted island motion and their energetics. The databases collected from adatom-island (2-8 atoms) diffusion characteristics for a large set of

homo- and hetero-epitaxial metallic systems (Cu, Ni, Pd and Ag) are used to extract a set of easily accessible features, geometrical and energetic, using physical insight which are then encoded. Those features along with activation energy barrier are used to train and test linear and non-linear statistical models. A non-linear model developed based on neural network technique predicts the diffusion energy barriers with high correlation with the calculated ones. In this talk, we present the results of kinetics study of these homo or hetero-epitaxial metallic systems some of whose barriers are used for training of the model and are compared to the corresponding quantities obtained from KMC simulation using energy barriers calculated from computationally intensive interatomic interaction potential based approach.

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Work supported in part by MMN-1710306.

9:20am **TF+AS+EL+PS-ThM5 Using Ellipsometry and XPS to Understand the Degradation of Thin-film Aluminum Mirrors Protected by Ultrathin Fluorides**, *M.R. Linford, Brian I. Johnson, R.S. Turley, D.D. Allred*, Brigham Young University

The LUVVOIR (Large, UV-optical-IR) telescope is a potential NASA flagship space-based observatory of the 2020's or 30's. It will utilize the largest mirrors ever put into space. The reflective coating for the mirrors will be aluminum, since there is no material with comparable reflectance at shorter wavelengths. However, to achieve high reflectance over the broadest energy range, the top surfaces of such Al mirrors must be protected against the formation of oxide layers that form quickly in air using wide-bandgap fluoride coatings, traditionally about 25 nm of MgF₂. Researchers have been endeavoring to use fluorides which are transparent further into the VUV (vacuum ultraviolet) like LiF and AlF₃, and to make these barriers more continuous by depositing them on heated surfaces and making the barriers thinner. However, when the barriers are thinner and when materials like LiF are exposed to moist air, degradation of VUV reflectance is observed. Thus, studying fluoride barrier-coated mirrors is vital. We have recently reported on the time dependent growth of apparent aluminum oxide thickness for two Al mirrors protected by ultrathin fluoride layers. These measurements were based on variable-angle, spectroscopic ellipsometric (VASE) measurements. (Allred, Thomas, Willett, Greenburg, & Perry, 2017) (Miles, 2017). VASE, however, does not provide chemical composition data. An independent analytical technique which is sensitive to surface composition is required. We have undertaken such investigations using X-ray photoelectron spectroscopy (XPS), and now report on correlations between optical properties and XPS for fluoride-coated aluminum mirror test structures.

9:40am **TF+AS+EL+PS-ThM6 Model for Amorphous Thin Film Formation and Validation**, *Rahul Basu*, VTU, India

A coupled set of equations describing heat and mass transfer during phase transformation is formulated. The model is extended to incorporate surface convective effects. These equations which are non linear due to the moving interface are linearized and decoupled. Effects of various heat transfer parameters are analyzed through small parameter expansions. Solutions obtained via this artifice allow closer examination of surface effects on the boundary layer of the phase transformation. A relation is found for the effect of the glass transition temperature versus the boundary layer thickness for several alloys in various groups of the Periodic Table. Earlier work and results are analyzed in light of the present analysis.

11:00am **TF+AS+EL+PS-ThM10 2D TMD Monolayer of MoS₂ BY ALD and Insight in the Mechanism by Surface Organometallic Chemistry**, *Elvje Alessandra Quadrelli*, CNRS CPE Lyon, France **INVITED**

Atomically-thin crystalline domains of MoS₂ [1] or WS₂ [2] are obtained from an organometallic amorphous deposit obtained by ALD/MLD.

This original result with respect to the state of the art has been mechanistically rationalized with in situ and in operando modelling studies on the oxide nanobeads at different annealing temperatures. This contribution will present the surface organometallic method, the characterization of the 2D layers (among which the first in-plane micrographs of ALD-grown MoS₂ samples)[1] and the proposed surface coordination chemistry mechanism at hand obtained with model studied on 3D silica beads. These model studies couple in operando infra-red spectroscopy, gas-chromatography detection of the released by-products and atomic composition of the deposit at each cycle, leading to molecular level understanding of the growth process.

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Acknowledgments : This work was carried out within the framework of the partnership between the C2P2 research unit (UMR 5265 CNRS CPE Lyon University Claude Bernard Lyon 1) and CEA's Directorate of Technological Research (DRT) on the nanochemistry platform installed in CPE Lyon. The authors of the papers below thank CPE Lyon, CNRS, CEA / LETI (Silicon Technology Department and nanocharacterization platform) for the support and the DRF / INAC for the collaboration in the framework of the "2D Factory" project.

Ref : [1] Cadot et al. *Nanoscale*, **2017**, 9, 467. [2] Cadot et al. *JSVT A* **2017**, 35, 061502.

11:40am **TF+AS+EL+PS-ThM12 A Novel Fourier Transform Ion Trap Mass Spectrometer for Semiconductor Processes**, *Gennady Fedosenko, H.-Y. Chung, R. Reuter, A. Laue, V. Derpmann, L. Gorkhover, M. Aliman, M. Antoni*, Carl Zeiss SMT GmbH, Germany

Real-time inline control of process gas compositions with high sensitivity has been of particular importance in recent years in the semiconductor industry and beyond. Commonly quadrupole residual gas analyzers (RGA) are used, together with Optical Emission Spectroscopy (OES) for process control and process development. However, most RGAs are not capable of measuring a whole mass spectrum fast enough to monitor etch or deposition processes of a few seconds. A new process control mass spectrometer, based on Fourier-Transform 3D Quadrupole Ion Trap technology, is more appropriate for real-time inline process monitoring.

The 3D-Quadrupole Ion Trap mass spectrometer *iTrap*[®] by ZEISS is installed in a vacuum chamber (~ 120mm x 120mm x 500mm) with a fast switching valve for pulsed gas sample injection (pulse duration ~ 50ms or less). An electron gun is used for ionization of the gas pules. The Ion Trap achieves ion trapping and accumulation by means of a radio frequency applied to the ring electrode of the trap. With the aid of advanced electronic amplifiers and selective ion excitation technique the ion oscillations can be measured electrically by means of the induced current on the cap electrodes without using any separate particle detector. The mass spectrum can be finally obtained in less than one second.

Real-time measurements of the hydrogen plasma cleaning process of Sn contaminated samples were performed with the *iTrap* mass spectrometer.

The working pressure of the plasma cleaning process was 0.5 mbar. Decreasing signal of SnH₄ and other contaminations from the samples which are directly correlated to the cleaning process were observed with *iTrap*. This result is extremely useful for the process control of plasma processes and inline real-time contaminations control for high-end applications.

Inline measurement at a MOCVD chamber showed that *iTrap* is capable to detect reaction products, contaminations on the wafer holder and dopant memory in real-time. These results demonstrate that *iTrap* is a very sensitive and fast process mass spectrometer suitable for real-time inline process monitoring.

Many etch processes take place in 10 to 30 s process steps. Different processes were examined with e.g. HBr or BCl₃ chemistry together with several wafer materials such as Silicon, Hafnium Oxide or Titanium Nitride. The obtained mass spectra show the etch plasma chemistry together with etch reaction products (HfCl_x, SiCl_x, etc.). This data gives new insight into the etch processes, which until now were rarely understood on a chemical level. First wafer effects related to the chamber cleaning and pre-coating steps prior to the etch step were also examined.

12:00pm **TF+AS+EL+PS-ThM13 Realization of Shifts in Threshold Voltage and Subthreshold Swing in Atomic Layer Deposited Zinc Oxide As Channel Layer through in-situ Half-Cycle Analysis**, *Harrison Sejoon Kim, A.T. Lucero, S.J. Kim, J. Kim*, University of Texas at Dallas

Thin film process monitoring of atomic layer deposition (ALD) has been adopted as the versatile technique to identify both chemical and physical properties of ALD films. Their *in-situ* characterization technique includes mostly Fourier-transform infrared spectroscopy, X-ray photoelectron spectroscopy, and quartz crystal microbalance analysis. [1-3] However, currently there are no reports on monitoring the results of sub-nm device physics even if we are already in the era of beyond 10 nm node semiconductor processes. Moreover, even if there are a few initial studies, demonstrating *in-situ* electrical characterization with ALD, it requires device packaging, which ultimately limits the flexibility to be further characterized. [4]

In this current work, we have developed an ultra-high vacuum (UHV) cluster tool equipped with thermal processing, plasma surface treatment, thin film deposition, and electrical characterization which can be

performed *in-situ* (Figure 1). With this feasibility, we demonstrate the deposition of semiconducting zinc oxide (ZnO) in inverted-coplanar structured thin film transistors (TFT). Diethylzinc (DEZ) and water (H₂O) is used as ALD precursors at 100°C. DEZ and H₂O half-cycle analysis is carried out to monitor the interface states of ZnO/dielectric (Figure 2). Initially, 45 ALD cycles of ZnO have shown switching behavior with an on/off ratio of ~10² in vacuum. Subsequent ALD cycle shifts the threshold voltage (V_{th}). V_{th} shifts associated with each ALD cycle are assumed to be attributed to the changes in interface trap density as a result of interface state passivation in ZnO during its growth, especially passivating fixed oxide charges (Q_{ox}). To understand interface states of ZnO and the bulk of oxide better, further analysis of shift of subthreshold swing (SS) is demonstrated. Since shifts in SS best represents changes in interface trap density, [5] it is worthwhile to note the changes in SS in metal-oxide-semiconductor transistors.

This work was supported by the Creative Materials Discovery Program on Creative Multilevel Research Center (2015M3D1A1068061) through the National Research Foundation(NRF) of Korea funded by the Ministry of Science, ICT & Future Planning.

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Thin Films Division

Room 104B - Session TF+AS+EL+EM+NS+PS+SS-ThA

IoT Session: Thin Films for Flexible Electronics and IoT

Moderators: Jesse Jur, North Carolina State University, Siamak Nejati, University of Nebraska-Lincoln

2:20pm **TF+AS+EL+EM+NS+PS+SS-ThA1 Ultraflexible Organic Electronics for Bio-medical Applications**, *Tomoyuki Yokota, T. Someya*, The University of Tokyo, Japan **INVITED**

Recently, flexible electronics has much attracted to realize bio medical application for their flexibility and conformability [1-3]. To improve these characteristics, reducing the thickness of the device is very effective [4]. We have developed ultra-flexible and lightweight organic electronics and photonics devices with few micron substrates. We fabricated the 2-V operational organic transistor and circuits which has very thin gate dielectric layers. The gate dielectrics were composed of thin aluminium oxide layer and self-assembled monolayers (SAMs). Due to the very thin substrate and neutral position, our device shows the highly flexibility and conformability. The device doesn't be broken after crumpling.

And also we fabricated highly efficient, ultra-flexible, air-stable, three-color, polymer light-emitting diodes (PLEDs) have been manufactured on one-micrometer-thick parylene substrates. The total thickness of the devices, including the substrate and encapsulation layer, is only three micrometers, which is one order of magnitude thinner than the epidermal layer of the human skin. The PLEDs are directly laminated on the surface of skin and are used as indicators/displays owing to their amazing conformability as their superthin characteristics. Three-color PLEDs exhibit a high external quantum efficiency (EQE) (12, 14, and 6% for red, green and blue, respectively) and large luminescence (over 10,000 candelas per square meter at 10 V). The PLEDs are integrated with organic photodetectors and are used as pulse oximeter.

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3:00pm **TF+AS+EL+EM+NS+PS+SS-ThA3 Molecular Surface Chemistry for Improved Interfaces in Organic Electronics**, *Jacob W. Ciszek*, Loyola University Chicago

Organic optoelectronic devices (OLED, OFETs, etc.) contain at least one, if not multiple instances of overlayers deposited onto organic semiconductors. The generated interface is inherently flawed with issues such as non-ohmic contact, overlayer delamination, or deposition induced damage arising. Traditionally, this is addressed by physical vapor deposition of yet another layer or by reengineering the materials in the device stack. In contrast, a reaction based approach allows for a wider range of function to be installed via molecular components in an organized and oriented manner, all while take advantage the inherent reactivity of the organic molecules which comprise the semiconducting layer. We have developed this approach via a "click-like" Diels-Alder chemistry whereby prototypical acene films (tetracene or pentacene) can be appended with a variety of small molecules to form an interfacial layer only ~5 Å thick. This chemistry is then applied towards improving the metal on semiconductor contact. As a demonstration of principle, Diels-Alder chemistry is utilized to form covalent bonds linking the organic semiconductor with a deposited metal contact thereby eliminating the poor adhesion present in this system. Application of the chemistry towards contact potential shifts is presented, while work towards sensing applications concludes the talk.

3:20pm **TF+AS+EL+EM+NS+PS+SS-ThA4 Investigation of Low Temperature ALD-deposited SnO₂ Films Stability in a Microfabrication Environment**, *Tony Maindron, S.M. Sandrez, N.V. Vaxelaire*, CEA/LETI-University Grenoble Alpes, France

For applications such as displays (LCD, OLED) or solar cells, it is mandatory to use Transparent Conductive Oxides (TCOs) so as to allow light to extract out of the circuit or for light harvesting, respectively. In display technology, TCOs are also studied so as to replace the silicon technology developed to make the semiconducting channel in thin-film transistors. The mainstream TCO today is indium tin oxide (ITO), (90% indium oxide and 10% tin oxide). It has the advantage of having a high conductivity and a low surface

resistivity, of the order of 10 ohm/square, while having a transmittance greater than 85% over the entire visible spectrum. However, it has several drawbacks, including the scarcity of indium and the high cost associated to its extraction process, which has prompted the scientific community to look for alternative TCOs. Among all TCOs, ZnO has been widely studied as a low cost single-metal oxide alternative material to ITO, as well as its multi-metal oxide derivatives using a dedicated metal dopant to control its electrical conductivity, as for instance with Al (ZnO:Al or AZO). One issue however with ZnO films lays in their relative chemical stability to water. It has been shown that films of ZnO or AZO change their physical properties when exposed to moisture: electrical conductivity decreases, roughness can increase, and optical characteristics are modified. By extension, when ZnO-based TCOs have to be finely patterned by photolithography, their high sensitivity to water-based solutions can be a severe issue. In our laboratory, we have noticed that ZnO and AZO films made by ALD at 150 °C typically are not stable in a microfabrication process: a large decrease (~ 20 %) of AZO thickness after the development step of photoresists used to pattern the TCO film has been noticed. One way to stabilize ZnO-based compounds is to anneal the films at high temperatures. However for some dedicated applications that use fragile substrates (plastic films for flexible organic electronics for instance), such post annealing processes at high temperature (typ. > 150 °C) cannot be applied. An alternative is to explore alternative materials to AZO: we found out that SnO₂ ALD (150 °C) shows very promising features regarding the stability under a microfabrication environment, while having good electrical and optical characteristics.

4:00pm **TF+AS+EL+EM+NS+PS+SS-ThA6 Dopant Distribution in Atomic Layer Deposited ZnO:Al and In₂O₃:H Films Studied by Atom Probe Tomography and Transmission Electron Microscopy**, *Y. Wu, B. Macco, Eindhoven University of Technology, The Netherlands; A.D. Giddings, T.J. Prosa, D.J. Larson, CAMECA Instruments Inc.; S. Kölling, P.M. Koenraad, F. Roozeboom, Erwin Kessels, M.A. Verheijen*, Eindhoven University of Technology, The Netherlands

Transparent conductive oxides (TCOs) are ubiquitous in many of today's electronic devices, including solar cells. Atomic layer deposition (ALD) is a promising method to prepare high quality TCO films due to its well-known virtues – i.e., precise growth control, excellent conformality and uniformity – combined with its damage-free character. Here we report on two types of TCO films that we have studied by a combination of atom probe tomography (APT) and high-resolution transmission electron microscopy (TEM). The aim was to get more insight into how the dopants are distributed in the films.

The first study was carried out on ZnO:Al prepared by alternating cycles of Zn(C₂H₅)₂ and H₂O with cycles of Al(CH₃)₃ and H₂O in a supercycle fashion at 250 °C. For this material it is known that the maximum conductivity achievable is limited by the low doping efficiency of Al. To better understand the limiting factors for the doping efficiency, the 3-dimensional distribution of Al atoms in the ZnO host material matrix has been examined at the atomic scale by the aforementioned techniques [1]. Although the Al distribution in ZnO films prepared by so-called "ALD supercycles" is often presented as atomically flat δ-doped layers, in reality a broadening of the Al-dopant layers was observed with a full-width-half-maximum of ~2 nm. In addition, an enrichment of the Al at grain boundaries was seen.

The second study involved In₂O₃:H prepared by InCp and a mixture of O₂ and H₂O at 100 °C. This material provides a record optoelectronic quality after post-deposition crystallization of the films at 200 °C. Since both the H dopant incorporation and the film microstructure play a key role in determining the optoelectronic properties, both the crystal growth and the incorporation of H during this ALD process were studied [2]. TEM studies show that an amorphous-to-crystalline phase transition occurs in the low temperature regime (100-150 °C), which is accompanied by a strong decrease in carrier density and an increase in carrier mobility. Isotope studies using APT on films grown with D₂O show that the incorporated hydrogen mainly originates from the co-reactant and not from the InCp precursor. In addition, it was established that the incorporation of hydrogen decreased from ~4 at. % for amorphous films to ~2 at. % after the transition to crystalline films.

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4:20pm **TF+AS+EL+EM+NS+PS+SS-ThA7 Roll-to-Roll Processable OTFT Sensors and Amplifier**, *Kai Zhang*, University of Oxford, Department of Materials, UK; *C.-M. Chen, B. Choubey, H.E. Assender*, University of Oxford, UK

The high flexibility and relatively low cost of organic electronics are gradually providing more possibility for their application. Compared with conventional silicon based electronics, organic electronics have relatively short lifecycles and processor speed, but they are more promising in the market of wearable and flexible devices, for example, wearable health care devices, simple memory devices and flexible displays. In recent years, some flexible and wearable sensors have been developed, e.g. skin-touching sensors embedded in a sports suit can detect the change of heart rate, blood pressure, ion concentration of perspiration, or infrared radiation from the human body.

In most sensors based on organic thin film transistors (OTFT) made to date, the semiconductors are employed directly to detect analytes. However, (1) the lifecycles of this design is short due to the low stability of organic semiconductors; and (2) any modification for selectivity needs to be compatible with the semiconductor.

In this paper, we present sensors based upon an extended floating gate in order to separate the sensory area from the semiconductor. Transistors are manufactured, using our roll-to-roll vacuum webcoating facility, using a high-throughput all evaporation process (Ding et al., 2016, Taylor et al., 2015). We have demonstrated the principle of operation of a floating gate sensor integrated with the vacuum-deposited OTFT, by means of a simple strain sensor, using ferroelectric PVDF on the extended floating gate to directly act as a sensory material. To amplify the sensor signal further, a series of current mirrors and differential amplifiers have been designed based on the properties of single OTFTs. The combination of organic amplifier and OTFT sensor will be helpful to transduce sensing signal to a suitable level for wireless signal reading from flexible devices.

Reference:

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4:40pm **TF+AS+EL+EM+NS+PS+SS-ThA8 Functionalization of Indium Gallium Zinc Oxide Surfaces for Transparent Biosensors**, *X. Du, S. John, J. Bergevin, Gregory Herman*, Oregon State University

Amorphous indium gallium zinc oxide (IGZO) field effect transistors (FETs) are a promising technology for a wide range of electronic applications including implantable and wearable biosensors. We have recently evaluated the functionalization of IGZO back channel surfaces with a range of self-assembled monolayers (SAM) to precisely control surface chemistry and improve stability of the IGZO-FETs. The SAMs evaluated include, n-hexylphosphonic acid (n-HPA), (3,3,4,4,5,5,6,6,6-nonafluorohexyl) phosphonic acid (FPA), and (3-aminopropyl) trimethoxysilane (APTMS). A comparison of the surface chemistry is made for bare and SAM functionalized IGZO back channel surfaces using X-ray photoelectron spectroscopy and electronic device measurements in air and phosphate buffer solution (PBS). We find significantly improved device stability with the SAMs attached to the IGZO back channel surface, both in air and PBS. We related this to the reduction of traps at the back channel surface due to SAM passivation. To further evaluate the IGZO-FETs as biosensors we have immobilized glucose oxidase (GOx) to the APTMS functionalized IGZO back channel surface using glutaraldehyde. We find that both the FPA functionalized and the GOx immobilized surfaces are effective for the detection of glucose in PBS. Furthermore, the GOx immobilized IGZO-FET based glucose sensors have excellent selectivity to glucose, and can effectively minimize interference from acetaminophen/ascorbic acid. Finally, we will discuss fully transparent IGZO-FET based glucose sensors that have been fabricated directly on transparent catheters. These results suggest that IGZO-FETs may provide a means to integrate fully transparent, highly-sensitive sensors into contact lenses.

5:00pm **TF+AS+EL+EM+NS+PS+SS-ThA9 Large Area Atmospheric Pressure Spatial ALD of IZO and IGZO Thin-film Transistors**, *C. Frijters, I. Katsouras, A. Illiberi, G. Gellinck, Holst Centre / TNO, Netherlands; Paul Poort, Holst Centre / TNO and SALDtech B.V., Netherlands*

Atmospheric pressure Spatial ALD is able to deliver high deposition rates while maintaining the advantages of conventional ALD, such as low defect density, high conformality and thickness uniformity. An emerging application for Spatial ALD is flat panel (OLED) display manufacturing. Examples include oxide semiconductors and dielectric layers for use in thin-film transistors (TFT's), and thin-film encapsulation for flexible OLED displays. As today's displays are fabricated on glass plate sizes in the order of several square meters, a remaining challenge is the development of large-area Spatial ALD deposition technology that is able to combine high throughput with uniform performance across very large areas.

We are developing large area Spatial ALD technology, and as a first step between the lab and the display fab, we have installed a large area Spatial ALD sheet-to-sheet tool which can handle up to 400x325 mm² sized substrates. With this tool we are able to deposit uniform films across a deposition width of 400 mm and thickness non-uniformities of ~ 1%. The whole tool is operated under an atmospheric pressure but inert N₂ environment. The tool can be used to deposit a variety of materials using both thermal and plasma-enhanced Spatial ALD.

We will present about the fabrication and performance of 30 cm x 30 cm TFT backplanes with InZnO_x (IZO) and InGaZnO_x (IGZO) oxide semiconductors deposited by spatial ALD. The IZO and IGZO films were deposited by plasma enhanced Spatial ALD using co-injected In-, Ga- and Zn-precursors and an atmospheric pressure N₂/O₂ plasma. The deposition process has been optimized in terms of film composition and electrical properties on a lab-scale reactor before being translated to the large area spatial ALD reactor. We will report on the yield and performance of the 30 cm x 30 cm TFT backplanes, including electrical properties such as the field effect mobility, V_{on} and bias stress stability and compare it with state-of-the-art sputtered IGZO TFT's. Finally, the challenges in up-scaling Spatial ALD to plate sizes of 1.5 m and beyond will be discussed.

5:20pm **TF+AS+EL+EM+NS+PS+SS-ThA10 Thin Film Ink-Jet Printing on Textiles for Flexible Electronics**, *Jesse Jur, I. Kim, H. Shahariar*, North Carolina State University

Inkjet printing of thin film flexible electronics on textiles is an emerging field of research with advances in wearable technology. In this study we describe for the first-time a reliable and conformal inkjet printing process of printing particle free reactive silver ink on textile surfaces. Reactive silver ink is printed on fibers with eclectic polymers ranging from polyester and polyamide, and different structures of textiles such as knitted, woven, and nonwoven fabrics. The conductivity and the resolution of the inkjet-printed tracks are directly related to the fiber structures in the fabrics. Multiple passes of printing layers are needed to confirm the percolation of the metal network on porous, uneven surfaces. The conformality and the electrical conductivity of the inkjet-printed conductive coating on PET textiles are improved by in-situ heat curing the substrate during printing and surface modification, for example, by atmospheric oxygen plasma treatments. The in-situ heat curing potentially minimizes wicking of the ink into the textile structures. We have achieved the minimum sheet resistance of 0.2 Ohm/sq on polyester knit fabric, which is comparable to the conductive thick-paste used in the screen-printed process, as well as other traditional physical and chemical deposition processes on textile fabrics/yarns. Additionally, we have constructed textile knit structures which changes electric percolation depending on structural deformations of the knit loops, providing positive and negative gauge factors upon stretching. The printed patterns are post-treated with diluted silicone/UV curable aliphatic water-soluble polyurethane coating to improve the durability during washing. These findings open up the possibility of integrating inkjet printing in the scalable and automated manufacturing process for textile electronic applications.

5:40pm **TF+AS+EL+EM+NS+PS+SS-ThA11 Flexography Oil Patterning for In-line Metallization of Aluminium Electrodes onto Polymer Webs: Commercial Roll to Roll Manufacturing of Flexible and Wearable Electronics**, *Bryan Stuart, T. Cosnahan, A.A.R. Watt, H.E. Assender*, University of Oxford, Department of Materials, UK

Vacuum metallisation of aluminium through shadow masks has been commercially used for decades for depositing electrodes on rigid semiconductor devices, however recent developments have enabled large area, continuous deposition of patterned metallization in the aesthetic printing industry and has the potential for electrode interconnects for

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devices on flexible substrates such as Polyethylene Terephthalate (PET) and Polyethylene Naphthalene (PEN). Flexible polymer webs of PET were coated (*See Supplementary Figure*) with 50 nm thick, 165 μm wide aluminium electrodes by commercially compatible manufacturing at roll-to-roll web speed of 2.4 m min^{-1} showing tremendous potential for large scale manufacturing of wearable electronic devices in transistors, low level energy generation (Thermoelectric generators), energy storage (thin film batteries) or display technologies (LED displays) [1].

The in-line patterning process relies on adaptation of flexography ink printing which typically transfers ink to a rubber patterned plate for printing images onto polymer webs. The flexibility of the printing plate accommodates for irregularities in the surface of the substrate making this process desirable for large area manufacturing. Metallization has been used to deposit aluminium onto a flexography applied pattern by replacing ink with a low vapor pressure oil (e.g. Perfluoropolyether, Krytox®). The radiative heating of aluminium metallization causes simultaneous evaporation of the oil pattern, thereby rapidly forming the desired metal pattern onto the un-patterned regions. Currently we are scaling-up the oil flexography/metallization process into an industrial-scale roll-to-roll coater with potential web widths of 350 mm and roll speeds of 100 m min^{-1} , in order to increase web speeds, and to expand the range of materials deposited and the functional devices to which they are applied.

This paper reports on in-line pattern deposition of aluminium and other materials as applied to functional devices, for example organic thin film transistors and thermoelectric devices. In particular we are able to demonstrate how the process can be compatible with other functional layers. We will report our studies of electrode precision (shapes/sizes) by patterned metallization and our first studies of sputtering with flexography patterning. The long term view is integration of this technology along the R2R production path for single pass/high speed production of low cost and flexible integrated circuits.

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