Spectroscopic Ellipsometry Focus Topic
Room: Cochiti - Session EL+AS+EM+MS+TF-ThA

Spectroscopic Ellipsometry

Moderator: A.M. Creatore, Eindhoven University of Technology, the Netherlands

2:00pm **EL+AS+EM+MS+TF-ThA1 Developments in Spectroscopic Ellipsometry for Characterization of Organic and Inorganic Surfaces, Interfaces and Complex Layered Materials**, ***M. Schubert***, University of Nebraska - Lincoln **INVITED**

In this paper we will review new developments in Spectroscopic Ellipsometry for characterization of organic and inorganic surfaces, interfaces and complex layered materials. Ellipsometry has matured over the past two decades with instrumentation and methodology capable of addressing today's and tomorrow's challenges in materials characterization and metrology. Ellipsometry measures the general state of polarization of light reflected or transmitted from samples. Owing to its nondestructive and generally applicable concept of investigating light emerging from specimens under investigations, and owing to its extreme accuracy and precision, ellipsometry has paved the way for almost all our modern technologies, continues to enable next-generation devices in electronics and optoelectronics, and emerges into fields of chemical, biochemical and biological platform technologies. Originating from the identification that the information carried upon the polarization within a light beam emerging from surfaces is extremely sensitive to mono and submonolayer characteristics, ellipsometry started decades ago pioneering development of microprocessors and electronic devices, which still today are based on planar thin film technology. Without ellipsometry, today's computation and information technology would be still in its infancy. In this paper, emerging developments and applications for metrology of optical and electrical properties of semiconductors and nanostructures by Terahertz Magnetooptic generalized ellipsometry, also referred to as the Optical Hall effect, will be highlighted. Examples will include state-of-the-art nitride semiconductor device structures and epitaxial graphene, candidates for tomorrow's next-generation devices. Likewise, new approaches for characterizing precise structural, magnetic and optical properties of three-dimensional nanostructure hybrid materials will be discussed. Examples will describe how ellipsometry characterization enables understanding and tailoring of electromagnetic properties of materials created by human intelligence, rather than by nature. New horizons are being explored currently by combining ellipsometry with independent surface sensitive techniques, such as acoustic Quartz-Crystal microbalance techniques. Combinations allow for identification of new information not accessible otherwise. Examples include observation of in-situ formation of self-assembled monolayers, protein adsorption onto sensitized surfaces, and formation of micelle-assisted bilayer configurations. Prospects, challenges and future developments will be reviewed from today's perspective.

2:40pm **EL+AS+EM+MS+TF-ThA3 Characterizing the Adsorption – Desorption Behavior of Organic Molecules Within Thin Mesoporous Carbon Composite Films using Spectroscopic Ellipsometry**, ***B.D. Vogt***, *L.Y. Song, M.Z. Dai*, Arizona State University

Porous carbon materials are commonly utilized as adsorbants (i.e. activated carbon) and as catalyst supports. Direct templated synthetic routes to form ordered mesoporous carbons have recently been developed. By utilizing these concepts, mesoporous carbon composite films containing metal oxides can be synthesized. As a wide range of metal oxides can be utilized, these materials could be utilized in chemical sensing applications or as catalysts in fuel cell membranes. For both of these applications, the porous material will be exposed to organic vapors (such as ethanol in fuel cells). The condensation and evaporation of organic vapors from these materials is therefore an important consideration for their ultimate utilization in these applications. In-situ spectroscopic ellipsometry measurements of the mesoporous films exposed to controlled vapor pressures of organic vapors such as toluene, hexane and ethanol are utilized to understand the adsorption-desorption behavior of these films. Activated desorption of all three compounds is observed for pure carbon films, but addition of a small fraction of metal oxide enables the organics to be desorbed for the porous framework. These adsorption-desorption isotherms can also be utilized to estimate the pore size distribution and porosity of these films.

3:00pm **EL+AS+EM+MS+TF-ThA4 Mueller-Matrix Ellipsometry Studies of Chirality in Chitin-Based Structures and Thin Films of $Al_{1-x}In_xN$**, ***K. Järrendahl***, *H. Arwin, R. Magnusson, P. Sandström, C.-L. Hsiao, J. Landin, S. Valyukh, J. Birch*, Linköping University, Sweden

A limited number of natural structures are known to reflect light that has circular or near circular polarization. This is for instance, the case for some scarab beetles were it is suggested that the polarization is caused by chiral structures in the form of helicoids in the cuticles . In this study, Mueller-matrix spectroscopic ellipsometry is applied in the spectral range of 250 to 1000 nm to investigate optical response and structures of the cuticle of various scarab beetles of the Cetoniinae subfamily. We will present our measurements showing how the polarization changes with wavelength as well as incidence angle and specify the conditions for when the reflected light is circularly left- or right-polarized. In most cases the reflected light is left-polarized as described by negative values of the M41 Mueller matrix element. For *Cetonia aurata*, a green beetle with metallic appearance, this is clearly seen in a rather narrow spectral range (470-550 nm). For other beetles (*Potosia cuprea* and *Licola lugubris*) similar polarization behavior is observed but the polarization features occur in a broader spectral region. We will show that there are even beetles (e.g. *Plusiotis argentiola*) reflecting both left- ($M41 < 0$) and right-polarized light ($M41 > 0$) in different parts of the spectral region. The Mueller data, including observations of the degree of polarization, are used to obtain structural and optical parameters from model calculations.

Our attempts to fabricate artificial structures with similar polarization properties will also be presented. $Al_{1-x}In_xN$ thin films were grown on sapphire substrates by magnetron sputtering of indium and aluminum in a nitride atmosphere. Utilization of different seed layers and a substrate rotation gave chiral structures constituted by layers with a compositional gradient. Mueller-matrix results from these structures will be compared with the results from the natural structures. The Mueller data is also in this case very rich on information. In the initial steps to model these samples a similar approach as for the natural structures has been employed. Similarities and differences of the natural and artificial polarization response will be discussed in detail.

3:40pm **EL+AS+EM+MS+TF-ThA6 Mueller Polarimetry as a Tool for the Evaluation of the Diffraction Grating Profile Asymmetry**, ***T. Novikova***, *P. Bulkin,* LPICM, CNRS, Ecole Polytechnique, France, *V. Popov,* Moscow State University, Russia, *A. De Martino*, LPICM, CNRS, Ecole Polytechnique, France

Mueller polarimetry in conical diffraction has proved to be a powerful optical technique for the metrological characterization of diffraction gratings. It was already shown that the shape of grating profile can be successfully reconstructed via appropriate optical modeling using full Mueller matrix measurements [1]. We also demonstrated that this approach can be of particular interest in microelectronics technology for the detection of overlay errors, which frequently result from the alignment deficiencies in lithography [2]. In some cases the asymmetrical distortion of grating profile can be induced by the etch process, or even be intentional, like in blazed gratings fabrication. For these applications a technique that allows for fast non-contact evaluation of the profile asymmetry may be of great value.

We studied the Mueller matrix spectra of symmetrical [3] and asymmetrical photoresist diffraction gratings on chromium using MM16 spectroscopic polarimeter, commercially produced by Horiba Jobin-Yvon, in the most general geometry of conical diffraction. At this configuration the 0th order cross-polarization complex reflection coefficients are antisymmetrical ($r^0_{sp} = -r^0_{ps}$) , provided that the grating is composed of only reciprocal materials and is invariant under the rotation by 180° about the normal incidence [4]. It leads to the following relations between the elements of 2x2 off-diagonal blocks of Mueller matrix: $M_{ij} = \pm M_{ji}$.

The lack of rotational symmetry violates the electromagnetic reciprocity theorem for the 0th-order diffraction on the asymmetrical gratings and, consequently, breaks the symmetry of the off-diagonal blocks of Mueller matrix ($|M_{ij}|$ is not equal to $|M_{ji}|$). This property of Mueller matrix of asymmetrical gratings was experimentally observed and numerically modeled at any illumination condition with exception of planar and pure conical mounting. We showed that the non-reciprocity in diffraction gratings can be used for the unambiguous detection of the grating profile asymmetry. The optimal choice of measurement configuration, i.e. azimuthal and polar angles considerably increases the sensitivity of the above mentioned technique.

References:

[1] T. Novikova, A. De Martino, S. Ben Hatit, and B. Drévillon, Appl. Opt. **45**, 3688 (2006).

[2] T. Novikova, A. De Martino, R. Ossikovski and B. Drévillon, *Europ. Phys. J. Appl. Phys.* **31,** 63 (2005).

[3] T. Novikova, A. De Martino, P. Bulkin, Q. Nguyen, B. Drévillon, V. Popov, and A. Chumakov, *Opt. Express* **15**, 2033 (2007).

[4] L. Li, Opt. Soc. Am. A **17**, 881 (2000).

4:00pm **EL+AS+EM+MS+TF-ThA7 Monitoring Ultra-Thin Organic Film Growth, *In-Situ*, with Combined Quartz Crystal Microbalance and Spectroscopic Ellipsometry**, ***K.B. Rodenhausen***, *B.A. Duensing, A.K. Pannier, M. Schubert,* University of Nebraska-Lincoln*, M. Solinsky,* The Procter & Gamble Company*, T.E. Tiwald*, J. A. Woollam Co., Inc.

We report a combinatorial approach to study ultra-thin organic films. This novel technique consists of *in-situ* spectroscopic ellipsometry and quartz crystal microbalance methods. In contrast to the quartz crystal microbalance, which is sensitive to the total mass attached to the surface, including the trapped solvent, spectroscopic ellipsometry only measures the amount of adsorbent on the surface. We also introduce a new "virtual separation approach" ($2\pi nd/\lambda \ll 1$) of analysis for the ellipsometry measurements. By using these two techniques in tandem, we are able to determine the thickness and solvent fraction of viscoelastic thin films.

We investigate cetyltrimethylammonium bromide (CTAB) thin films deposited onto a gold-coated quartz crystal as a model system. CTAB grown from a 2.5 mM solution demonstrates several phases in porosity evolution, including a temporary hold in water fraction as the film is rinsed off the substrate with water; these effects may be related to the structure of a CTAB bilayer.

In addition, a variety of self-assembled monolayers (SAMs) of alkanethiols on gold-coated quartz crystals are used as model biomaterials to determine the water fraction of an adsorbed prion layer. The porosity information distinguishes the proteins' conformation, dictated by the defined surface chemistries of the SAMs.

4:20pm **EL+AS+EM+MS+TF-ThA8 Ellipsometric Studies of Electronically Coupled PbSe and PbS Quantum Dot Thin Films**, ***S.G. Choi***, National Renewable Energy Laboratory*, O.E. Semonin,* University of Colorado*, J.M. Luther, M.C. Beard, A.G. Norman,* National Renewable Energy Laboratory*, Z. Lin,* Colorado School of Mines*, A. Franceschetti,* National Renewable Energy Laboratory*, M.T. Lusk,* Colorado School of Mines*, A.J. Nozik*, National Renewable Energy Laboratory

Discovery of multiple exciton generation from colloidal suspensions of semiconductor quantum dots (QDs) has generated growing interests in realization of high-efficiency QD-based solar cells. Among a number of semiconductor QDs explored up to date, lead chalcogenides such as PbSe and PbS have been of great interest as a result of their wide tuning range of bandgap energy, abundance of materials, and large exciton Bohr radius.

In this presentation, I discuss optical properties of electronically coupled PbSe and PbS QD thin films. A series of QD multilayer thin films were prepared by a layer-by-layer dip-coating method onto glass substrates. Diameter of the QDs varies from 3.2 to 7.2 nm and from 3.5 to 8.3 nm for PbSe and PbS, respectively. Room-temperature pseudo-optical functions of the samples were measured by a rotating compensator-type, variable-angle spectroscopic ellipsometer. Transmittance data were also acquired in a normal-incidence configuration.

First, I determined refractive index $N = n + ik$ of the QD films using the B-spline basis functions within the multilayer model (ambient/surface roughness/QD film/substrate). We use the N obtained as the input parameters for modeling the internal quantum efficiency of the QD-based solar cell devices. Then, I extracted dielectric function $\varepsilon = \varepsilon_1 + i\varepsilon_2$ for the ensemble of electronically coupled QDs using the Maxwell-Garnett effective medium approximations. The ε spectra show the first exciton peaks, and the E_1 and E_2 critical-point (CP) structures whose energies are higher than the corresponding bulk values probably due to the quantum confinement effects. This abstract is subject to government rights.

4:40pm **EL+AS+EM+MS+TF-ThA9 In-situ Temperature Measurements by Spectroscopic Ellipsometry: Application to a-Si based Thin Films**, ***D. Daineka***, LPICM, CNRS, Ecole Polytechnique, France*, V. Suendo,* Institut Teknologi Bandung, Indonesia*, P. Roca i Cabarrocas*, LPICM, CNRS, Ecole Polytechnique, France

Accurate measurement of the substrate temperature is of crucial importance in many semiconductor technologies such as plasma enhanced chemical vapor deposition (PECVD). Traditional tools, both thermocouples and pyrometers, are not always reliable for in situ measurements in vacuum when the substrate can be out of thermal equilibrium. On the other hand, non-contacting optical methods allow to determine the surface temperature with great accuracy, provided the temperature dependence of optical constants for the studied material is known. Since recently, spectroscopic ellipsometers are widely available and often installed on the research deposition systems, which provides an opportunity to use them for temperature monitoring. We have studied the optical functions of amorphous silicon based thin films with spectroscopic ellipsometry in the temperature range from 290 to 520 K. The experimental data were modeled using Tauc-Lorentz dispersion law for amorphous materials. We have found that the temperature coefficients of Tauc-Lorentz parameters, such as the optical gap, are rather close for a few different materials. That similarity suggests that these values can be used to determine the surface temperature for a broad range of amorphous silicon based materials with a good accuracy. Practical examples of using spectroscopic ellipsometry for temperature measurements in the low pressure PECVD environment are given.

5:00pm **EL+AS+EM+MS+TF-ThA10 Real Time Spectroscopic Ellipsometry Studies of Amorphous and Nanocrystalline $Si_{1-x}Ge_x$:H Thin Films for Microbolometer Applications**, ***D.B. Saint John***, *H.-B. Shin, M.-Y. Lee, E.C. Dickey, T.N. Jackson, N.J. Podraza*, Penn State University

Hydrogenated amorphous and nanocrystalline silicon (a/nc-Si:H), germanium (a/nc-Ge:H), and their alloys have been used and continue to be assessed for use in uncooled infrared microbolometer applications. These materials may be deposited as uniform layers using equipment common to the manufacturing of displays and photovoltaics and are thus more amenable to manufacturing considerations than the ion beam deposited vanadium oxide films used in most commercial microbolometers. Real optimization of material in the a/nc-$Si_{1-x}Ge_x$:H system for use in these devices requires a better understanding of the relationship between the key electrical properties of interest including resistivity (r), temperature coefficient of resistance (TCR), and the 1/f noise character as a function of the degree of order and composition of the films. $Si_{1-x}Ge_x$:H thin films were deposited using plasma enhanced chemical vapor deposition using SiH_4, GeH_4, and H_2 at variable H_2- dilution. These films have been monitored using in situ real time spectroscopic ellipsometry (RTSE) over a spectral range from 0.75 to 5.15 eV during deposition to detect changes in the film thickness and optical properties in the form of the complex dielectric function spectra ($e = e_1 + ie_2$) as a function of deposition time. From the RTSE measurements and analysis it is possible determine the structure of the material as amorphous, nanocrystalline, or mixed-phase and track the evolution of nanocrystallinity as a depth profile into the film. Ex situ Fourier transform infrared spectroscopic ellipsometry measurements over a spectral range from 0.05 to 0.75 eV were also performed to augment the complex dielectric function spectra and study absorption features relating to bonding. For electrical measurements, contacts were deposited in an isolated transfer length pattern for measurement of resistivity and TCR, while resistors with different volumes were made for volume normalization of the 1/f noise measurements. The TCR was measured from 20°C to 55°C. This study explores to correlations between the electrical and optical properties of a-$Si_{1-x}Ge_x$:H and nc-$Si_{1-x}Ge_x$:H as functions of film processing conditions, resultant composition, and order. a-$Si_{1-x}Ge_x$:H films were prepared as a function of germanium content and hydrogen dilution to identify the impact that germanium and improved order at higher hydrogen dilution conditions have on the electrical properties (r, TCR, 1/f noise). The impact on the electrical properties due to the incorporation of small fractions of nanocrystallites are explored using mixed-phase (a+nc)-Ge:H films with nanocrystallite profiles guided by depth profile studies.

5:20pm **EL+AS+EM+MS+TF-ThA11 Roll-to-Roll Fabrication of Thin Film Si:H Solar Cells: Real Time Monitoring and Post Deposition Mapping by Spectroscopic Ellipsometry**, ***L.R. Dahal***, *Z. Huang, D. Attygalle, M.N. Sestak, C. Salupo, S.X. Marsillac, R.W. Collins*, University of Toledo

Real time spectroscopic ellipsometry (RTSE) has been used to monitor the roll-to-roll deposition of thin film Si:H n-i-p solar cells on flexible plastic substrates coated with a Ag/ZnO back-reflector. In this process, the RTSE monitoring position is located directly above the ZnO sputtering target (i.e., at the closest target-substrate separation). RTSE data collection is initiated before the plasma is ignited so that ZnO nucleation can be observed. The film thickness increases with time until a steady state is reached, after which the bulk layer thickness at the monitoring point is constant with time. This occurs when the elapsed deposition time equals the time required for the moving substrate to travel from the leading edge of the deposition zone to the monitoring point. Although a constant substrate speed is selected such that the final film thickness is achieved in the time required to move through the entire deposition zone, this speed does not allow study of film growth that occurs after the substrate passes the monitoring point. To solve this problem, the substrate speed is reduced only in the early stage of growth such that the final film thickness of interest is reached at the monitoring point. In this way, RTSE can be used to analyze the entire layer on an initial length of the roll before the full length of the roll is processed. The thickness evolution of ZnO in the case of both normal and reduced

speeds shows good agreement with a simple inverse square variation of the deposition flux from the target to the flexible substrate.

After cell deposition, spectroscopic ellipsometry (SE) has also been applied for large area mapping of the completed 15 cm wide roll, at up to 1.5 m long sections at a time. Key information such as critical point, oscillator amplitudes, band gap energies, and widths have been extracted from which material density, composition, grain structure, disorder, and defect density can be determined. In this paper, optical mapping was applied for the intrinsic absorber layer in a full device a-Si:H solar cell structure. The results clearly show the degree to which thickness uniformity of the absorber layer depends on the gas flow and the electrode configuration. Also, by parameterizing the optical functions of the intrinsic absorber layer using single Lorentz oscillator modified by a low energy absorption cut-off, a map of its band gap and oscillator width can be deduced. Such an SE application is ideal for evaluation of uniformity in bulk thickness db, surface roughness thickness ds, index of refraction, and extinction coefficient (n, k); the critical parameters for fabricating uniform and high efficiency solar modules.

Spectroscopic Ellipsometry Focus Topic
Room: Southwest Exhibit Hall - Session EL+AS+EM+MS+TF-ThP

Spectroscopic Ellipsometry Focus Topic Poster Session

EL+AS+EM+MS+TF-ThP1 Temperature Dependence of the Dielectric Function of AlSb Measured by Spectroscopic Ellipsometry, ***J.J. Yoon***, *Y.W. Jung, J.S. Byun, S.Y. Hwang, Y.D. Kim,* Kyung Hee University, Republic of Korea*, S.H. Shin, S.Y. Kim, J.D. Song*, Korea Institute of Science and Technology, Republic of Korea

AlSb is a promising material for applications in heterostructure devices such as long-wavelength detectors, quantum-well lasers, and laser diodes. However, to understand and properly design these devices, information about its electronic properties and its dielectric function $\varepsilon = \varepsilon_1 + i\varepsilon_2$ is needed. While room-temperature ε data for AlSb exist, very little information is available about its behavior at elevated temperatures. Here, we report pseudodielectric function data $\langle\varepsilon\rangle$ from 300 to 800 K and from 0.7 to 5.0 eV, determined by spectroscopic ellipsometry. The samples were 1.5 µm thick layers grown on GaAs (001) substrates by molecular beam epitaxy (MBE). This thickness significantly exceeds the critical value for AlSb, so the layers are fully relaxed. The MBE station features an integrated spectroscopic ellipsometer and strain-free windows, thereby allowing e data to be obtained without exposing the samples to air. For AlSb this is critical, because the removal of its oxides is not feasible owing to its reactivity. As a result of these precautions and the method by which these $\langle\varepsilon\rangle$ data were obtained, we consider them to be the most accurate representation of ε to date. We also analyzed these data for critical-point (CP) parameters by fitting numerically calculated second energy derivatives of to standard analytic CP lineshape expressions. A parametric model was used, which describes dielectric functions by a combination of energy-bounded polynomials and poles, and encodes information in terms of amplitudes, critical-point energies, and broadening parameters. The reconstructed spectra are in excellent agreement with the data. We use these parameters to obtain information about the individual oscillators, including phonon effects, and interpolate them to obtain an analytic representation of the dielectric response of AlSb as a function of temperature. We expect these results to be an important database supporting engineering design, device technologies, and in-situ monitoring and control of device fabrication.

EL+AS+EM+MS+TF-ThP2 Optical Properties and Humidity Effects on Thin Films of Micro Fibrillated Cellulose Studied by Spectroscopic Ellipsometry, *H. Arwin,* ***E. Antunez de Mayolo***, Linköping University, Sweden*, M. Eita,* Royal Institute of Technology (KTH), Sweden*, H. Granberg,* Innventia Ab, Sweden*, L. Wågberg*, Royal Institute of Technology (KTH), Sweden

High quality micro fibrillated cellulose (MFC) prepared from wood fibers can be used to prepare thin films on solid substrates by a layer-by-layer deposition technique. In applications MFC layers can be used alone or as a constituent in functional coatings, where the MFC can make a significant contribution to mechanical properties of the coating. Examples of potential applications are sensor layers, decorative coatings or mirrors. Here the optical properties in terms of the refractive index of MFC are studied with *in situ* spectroscopic ellipsometry (SE) and the effects on thickness and index due to humidity are investigated.

Films of MFC and polyethyleneimine (PEI) in the thickness range 30 - 300 nm are deposited on silicon substrates. Such MFC/PEI samples are exposed to water vapor in nitrogen (0-90% RH) and the ellipsometric response are measured *in situ* in the spectral range 245 – 1700 nm at an angle of incidence of 70 degrees using a dual-rotating compensator ellipsometer. From the SE-data the MFC/PEI film thickness and layer index are modeled.

Due to water exposure, the thickness of an MFC/PEI film is found to increase up to 15% or more, whereas the refractive index decreases. The effects are fully reversible and the dynamics of these changes are monitored with SE and are discussed.

EL+AS+EM+MS+TF-ThP3 Spectroscopic Ellipsometry and X-ray Photoelectron Spectroscopy of La_2O_3 Thin Films Deposited by Reactive Magnetron Sputtering, ***V. Atuchin***, Institute of Semiconductor Physics, Russia*, A.V. Kalinkin,* Boreskov Institute of Catalysis, Russia*, V.A. Kochubey, V.N. Kruchinin,* Institute of Semiconductor Physics, Russia*, R.S. Vemuri, C.V. Ramana*, University of Texas at El Paso

Lanthanum trioxide (La2O3) is one among the most promising high-*k* dielectric materials to replace SiO2 and Si3N4 in advanced metal-oxide-semiconductor devices in gate stack. La2O3 can be prepared by various techniques but the film properties are strongly dependent on the fabrication conditions. Reactive magnetron sputtering deposition is widely used for the preparation of high quality transition multivalent metal oxide films with reproducible parameters and controlled thickness. The technique is preferred since it usually offers a high deposition rate for oxide films and a possibility to control the chemical composition of the film by reactive atmosphere in vacuum chamber. The aim of the present study is to understand the surface structure and evaluate the optical parameters of La2O3 films deposited on Si substrates by magnetron sputtering. La2O3 thin films were deposited onto Si(100) substrates in an argon/oxygen atmosphere using a high purity La target (99.9%). Structural parameters of the films were estimated by reflective high energy electron diffraction (RHEED) method at electron energy of 50 keV. All the films show no diffraction pattern indicating their amorphous nature in the near surface layers. Chemical state examined by the X-ray photoelectron spectroscopy (XPS), SPECS device, monochromatic Al Ka radiation (1486.6 eV) before and after Ar+ (2.5 keV, 2 min) sputtering indicates the stoichiometric film formation. Chemical nature of the species was identified with using binding energy (BE) difference parameter DLa = BE (La 3d5/2) - BE (O 1s) [1]. Optical parameters of the films were measured with spectroscopic ellipsometry (SE) using a Spectroscan ellipsometer in the spectral range of 250 nm < l < 1100 nm at the incidence angle of 70°. The La2O3 films with thickness 500-850 nm were transparent over the spectral range and dispersion relations of refractive index *n* were well derived using a model of (air)/(single homogeneous layer)/(Si substrate). The curves *n*(l) were approximated by Caushy polynomials. Good relation between experimental points and theoretical curves confirms applicability of this simple model for the films. The optical parameters of La2O3 defined with SE are related to film bulk and are insensitive to top surface effects induced by hydration or carbonate formation. These parameters will be used as a basis for SE evaluation of nanometric lanthanum oxide films with thickness below 10 nm.

1.V.V. Atuchin, T.A. Gavrilova, J.-C. Grivel, V.G. Kesler, Electronic structure of layered ferroelectric high-k titanate La2Ti2O7, J. Phys. D: Appl. Phys. 42 (2009) 035305.

EL+AS+EM+MS+TF-ThP4 Analysis of Anomalous Film Growth when Yttrium Oxide Thin Films are Exposed to 7.2eV Light, ***D. Mortensen***, *D.D. Allred*, Brigham Young University

We have recently found that exposure of reactively sputtered yttrium oxide thin films to 7.2 eV photons in air produces a dramatic (factor of 4) increase in the films' thickness. This result was completely unexpected, Y2O3 is exceptional stable, and demanded further investigation. This is particularly important since yttria and neighboring metal oxides such as ZrO2 have been considered, and HfO2 is being used, as components in gate oxides for silicon devices. The excimer lamp used for the study was of the type used in cleaning the surfaces of silicon wafers in the semiconductor industry. It is vital to understand how metal oxides might swell during cleaning.

We have experimentally observed the following:

1. The film-thickness increase is linear with exposure time up to a point.

2. Over a factor of four increase in film thicknesses, as measured by spectroscopic ellipsometry, has been observed. E.g., film which was about 20 nm thick as deposited reached a thickness of about 100nm.

3. When the yttrium oxide sample is placed in a furnace the changes rendered to the yttrium oxide sample are reversed.

4. A film placed in a plasma cleaner does not show this increase.

5. These effects are noticeably absent under the same conditions for a silicon wafer.

6. The refractive index of the film decreased with exposure time, suggesting the film could becoming more porous, though effective media optical models were unsuccessful in modeling the optical properties.

In addition to ellipsometry the films were studied by XPS and STEM.

EL+AS+EM+MS+TF-ThP5 In-Situ Spectroscopic Ellipsometry of Lithium Ion Intercalation in GLAD Three-Dimensional Nanostructured Thin Films, ***E. Montgomery***, *M. Schubert, E.B. Schubert, T. Hofmann, D. Schmidt,* University of Nebraska - Lincoln, *R.A. May*, University of Texas at Austin

Lithium intercalation in thin nanostructured and bulk films from metals and semiconductors has been studied using in-situ spectroscopic ellipsometry. Nanostructured thin films have a large surface area, and the stress caused by the intercalation of lithium is reduced in comparison to continuous films. The films are deposited using electron beam evaporation at a glancing angle or Glancing Angle Deposition (GLAD).

The charge capacity of the films can be observed as a change of index of refraction using ellipsometry. We will study intercalation as a function of structure geometry in chiral and achiral 3D GLAD thin films deposited from different metals onto silicon and other dielectric substrates. We report the anisotropic dielectric functions of the 3D nanostructures and their changes as a function of intercalation time and period.

EL+AS+EM+MS+TF-ThP6 Multi Phase Model Generation of Reflection Anisotropy Spectra of Copper Phthalocyanine Films on Vicinal Silicon Substrates, ***F. Seidel***, *L. Ding, O.D. Gordan, D.R.T. Zahn*, Chemnitz University of Technology, Germany

In this work the in-plane anisotropy of copper phthalocyanine (CuPc) thin films grown on vicinal silicon substrates is explained by simulating Reflection Anisotropy Spectroscopy (RAS). In RAS the complex difference in reflection along two perpendicular directions is measured at an incidence angle close to 0°. While RAS has strong similarities with another polarisation related measurement technique, Spectroscopic Ellipsometry (SE), simulations of RA spectra using a similar mathematical formalism like in SE is not common.

One difference is that in SE the measurements are usually performed at an incidence angle close to the Brewster angle, where the difference between s and p reflected polarisation is maximal. However due to the similarities between the two techniques the evaluation of RA spectra can be performed in a similar way like for SE after some simple mathematical transformations. This in particularly useful when thickness induced interference can lead to incorrect interpretations of RA spectra. Therefore in this work we show that the RA spectra evaluation of CuPc layer with increasing thickness is mainly given by the optical interference effect and not by a change in the optical anisotropy of the film.

Spectroscopic Ellipsometry Focus Topic
Room: Cochiti - Session EL+AS+EM+MS+TF-FrM

Spectroscopic Ellipsometry - Inorganic Thin Films
Moderator: L.A. Giannuzzi, L.A. Giannuzzi & Associates LLC

8:20am **EL+AS+EM+MS+TF-FrM1 Spectroscopic Ellipsometry Study on Transparent Conductive Ga-doped ZnO Thin Films Deposited by Ion-Plating with DC Arc Discharge**, *T. Yamada,* ***H. Makino****, N. Yamamoto, T. Yamamoto*, Kochi University of Technology, Japan

Transparent conductive Ga-doped ZnO (GZO) thin films was one of promising candidates as transparent electrodes in flat panel displays and thin film solar cells. In this study, we investigated contributions of intra-grain scattering and grain boundary scattering on Hall mobility of polycrystalline GZO thin films based on the analysis of spectroscopic ellipsometry measurements. The GZO films were deposited by ion-plating method with DC arc discharge. Samples with a wide range of thicknesses from 70 to 500 nm were deposited at 200 degree C on glass substrates by controlling the deposition time. Sintered ZnO ceramic tablets doped with 1 wt% and 4 wt% Ga_2O_3 were used as evaporation sources. A systematic study has been done on the structural, electrical and optical properties of GZO films. In the case of GZO films using 4wt% Ga_2O_3 doped tablet, the minimum resistivity of 1.8×10^{-4} Ωcm was obtained at the film thickness of around 350 nm. The carrier concentration and Hall mobility was 1.2×10^{21} cm^{-3} and 29 cm^2/Vs, respectively. The GZO films showed c-axis preferential orientation nearly normal to the substrate surface with columnar grain structures. In XRD measurements, full width half maximum of (0002) omega rocking curve decreased with increasing the film thickness. It means that the fluctuation in the c-axis orientations among the grains improved with increasing the film thickness. Average grain size of columnar structure was estimated by Williamson-Hall plot on spectral width of in-plane XRD patterns. The average grain size increased with increasing film thickness. Optical mobility of the GZO films was estimated from analysis using the conventional Drude model on data obtained by spectroscopic ellipsometry. Differences between the optical mobility obtained by the spectroscopic ellipsometry and the Hall mobility obtained by Hall effect measurements suggest contribution from grain boundary scattering on the Hall mobility. It demonstrates that grain boundary scattering contributes to the Hall mobility in rather thinner film thickness. However, the very small contribution of grain boundary scattering to the Hall mobility was observed in the thick films. On the other hand, resistivity of 4.5×10^{-4} Ωcm with the carrier concentration of 3.6×10^{20} cm^{-3} and Hall mobility of 39 cm^2/Vs was obtained for a 500 nm thick GZO film using the ZnO ceramic target doped with 1 wt% Ga_2O_3. The spectroscopic ellipsometry analysis on the GZO film suggests that the Hall mobility was dominated by intra-grain scattering.

8:40am **EL+AS+EM+MS+TF-FrM2 Spectroscopic Ellipsometry of Pulsed Laser Deposited ZnO on Atomic Layer Deposited Al_2O_3 and HfO_2**, ***D.L. Agresta****, K.D. Leedy*, U.S. Air Force Research Laboratory

Zinc oxide thin films and coatings are an integral part of a number of devices because of their wide range of properties. Depending on the conductivity and transparency, these materials are of interest for electronic (diodes, FETs), optoelectronic (detectors, modulators, flat-panel displays, solar cells, etc.) and piezoelectric (BAW,SAW) devices. In this study, we examine FET ZnO active device layers in conjunction with atomic layer deposited (ALD) Al_2O_3 and HfO_2 for isolation and gate dielectrics. Stacks of pulsed laser deposited (PLD) ZnO on thermal and plasma ALD Al_2O_3 and HfO_2 are prepared on a variety of large area substrates.

The surface morphologies of PLD grown ZnO-based films and ALD dielectrics are critical to optimize the interface quality in multilayer structures. Therefore, the control and optimization of surface properties over large areas is essential for the successful application of PLD ZnO thin films in device configurations. We have previously reported high quality ZnO films being obtained over large areas using off-axis PLD [1, 2]. Film thickness and compositional uniformity control is a critical issue associated with the scale-up of PLD systems. Thus, spectroscopic ellipsometry (SE) has a significant role in the characterization of these films.

A commercial Phase-Modulated Ellipsometer (PME) is employed to study the interplay between structural, electrical and optical properties of single-layers and PLD ZnO/ALD dielectric stacks. Known for its precision and non-destructiveness, SE is an indirect measurement technique in the sense that the film properties of interest are obtained by a nonlinear regression analysis of measured data to an optical model. We examine the effect on the goodness of fit parameter from the use of various optical models. Furthermore, since we are employing a PME, the time to scan a wide spectral region is limited by the slew rate of the monochromater. This can be a significant burden when making uniformity determination over large-area substrates. Thus, we examine the effect on the goodness of fit parameter from minimizing data acquisition time. The optical properties of the individual thin films extracted by SE are compared to optical studies using normal incidence transmission. Film surface roughness is obtained from SE by employing the Bruggeman effective-medium approximation and subsequently compared to structural characterizations using atomic force microscopy and x-ray diffraction.

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9:00am **EL+AS+EM+MS+TF-FrM3 Processing and Stability Studies of Vanadium Oxide Thin Films for Microbolometer Applications**, ***M.A. Motyka****, B.D. Gauntt, E.C. Dickey, M.W. Horn, N.J. Podraza*, Penn State University

Vanadium oxide (VO_x) thin films are commonly used as an imaging material in uncooled infrared sensing devices. Material properties that make VO_x useful for this application are a high temperature coefficient of resistance (TCR), controllable resistivity (ρ), and low electrical noise. A difficulty in growing VO_x thin films arises from the many valence states of vanadium, which may result in formation of a film consisting of an undesirable phase or with the presence of multiple phases. Each phase has varying electrical properties and thus, the reliability and consistency in industrial fabrication is lowered. Furthermore, atmospheric exposure of the VO_x films has been shown to alter the electrical and optical properties. In order to prevent changes in the desired material, VO_x films are commonly capped with a thin layer of SiO_2 before atmospheric exposure. In this study, vanadium oxide thin films were studied using in situ real time spectroscopic ellipsometry (RTSE) over a spectral range of 0.75 to 5.15 eV during deposition via pulsed DC-magnetron sputtering in an argon and oxygen atmosphere, with the set of variables being the total pressure, the oxygen-to-argon ratio, target power and the target material (metallic V, VO_2, V_2O_5). These variables control the material growth and resulting optical and electrical properties. The growth evolution, complex dielectric function spectra ($\varepsilon = \varepsilon_1 + i\varepsilon_2$), and structure obtained from RTSE have been shown to correlate with the electrical properties of the film. Ex situ Fourier transform infrared spectroscopic ellipsometry (FTIR-SE) measurements were also made to help characterize the materials in the spectral range of 0.05 to 0.75 eV, so that the optical properties in the range of microbolometer operation are obtained. Electrical measurements include temperature dependent I-V curve measurements to determine the VO_x film resistivity and TCR as a function of processing conditions. Changes in the optical and electrical properties as a function of processing conditions including film thickness are explored for materials exhibiting amorphous or nanocrystalline (V, V_2O, and VO phase) structures. RTSE is also used to monitor the changes in optical properties of the VO_x layer and interfacial formation arising from the deposition of the SiO_2 capping layer. The environmental stability of VO_x with and without capping layers is also monitored via RTSE as the samples are initially exposed to the atmosphere after deposition. In this manner both intentional variations in film microstructure and electrical properties as a function of processing conditions and unintentional variations arising from material instability are studied.

9:20am **EL+AS+EM+MS+TF-FrM4 Instrumentation of Far-infrared Mueller Matrix Ellipsometer and Its Application for Multiferroic Materials**, ***T.D. Kang****, P.D. Rogers, E. Standard, G.M. Nita, T. Zhou,* New Jersey Institute of Technology*, G.L. Carr,* Brookhaven National Laboratory*, S. Zollner,* IBM Systems and Technology Group*, M. Kotelyanskii,* Rudolph Technologies, Inc.*, A. Sirenko*, New Jersey Institute of Technology

We develop a far-IR spectroscopic ellipsometer at the National Synchrotron Light Source (NSLS) in Brookhaven National Laboratory (BNL). This Ellipsometer is designed to measure a full-Mueller matrix of the sample by using compensators and wire-grid linear polarizers [1]. With the exceptional brightness of synchrotron radiation and the Fourier-transform infrared (FT-IR) spectrometer, we measure ellipsometric data with a high accuracy at multi-wavelengths between 10 and 4,000 cm^{-1}. Study in terms of temperature variation of the sample is available using the cryostat for temperature between 4.2 K and 450 K. The wide range of θ-2θ rotation, c angle adjustment, and X-Y-Z translation of sample stage enables high accuracies in the alignment, calibration, and ellipsometric measurement.

With the Labview program interface, the automated experiments with the pre-programmed measurement schedules are performed by controlling the motors, temperature, and FT-IR spectrometer. The ellipsometric data analysis is based on the Berreman 's 4×4 propagation matrix formalism [2] to extract dielectric permittivity and magnetic permeability tensors for bulk and thin film samples from the Mueller matrix measured at variable incidence angles and sample orientations across the broad far-IR spectral range. Applications of this far-IR ellipsometry for multiferroic materials with $\mu \neq 1$ will be discussed. This development effort is supported by NSF-MRI-0821224.

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9:40am **EL+AS+EM+MS+TF-FrM5 Roughness beyond Bruggeman's Effective Medium Approximation**, ***H. Wormeester***, University of Twente, Netherlands **INVITED**

Surface roughness is regularly characterized with ellipsometry, which is especially sensitive for the short length scale roughness. Because of this, the roughness can be treated as a heterogeneous material modelled with an Effective Medium Approximation (EMA). The EMA layer thickness determined is often successfully related to the root mean square roughness from microscopy. A breakdown of this correspondence was recently shown [1]. This was attributed to the non-negligible influence of the characteristic length scale of the roughness. This typical characteristic length scale can approach the wavelength of the light used for many cases of surface roughness. In thus violates an important prerequisite of EMA, i.e. a variation limited to a length scale much smaller than the wavelength of light. This not only results in off-specular scattering, but also to a change in the polarization of the specular reflected light beam as probed with ellipsometry.

The applicability of an EMA to describe small surface roughness can be evaluated with the Rayleigh-Rice (RR) perturbation. In this perturbation method, the surface roughness is incorporated via its power spectral density function. Ohlidahl and co-workers [2] extensively compared Gaussian roughness distributions with EMA results. They reported that Bruggeman's equation describes the roughness well in many situations. However, the correspondence between EMA and RR breaks down for surface heterogeneity if noble metals are involved. For example for deposited colloids, the resonance energy of the induced surface plasmon is not correct [3]. Also the optical spectra calculated with EMA for a rough silver surface can only be reproduced by RR if a very specific power spectral density is used, showing a quite large characteristic length scale.

Roughness with various length scales created by oblique incidence ion sputtering on Ag(001) were experimentally studied with normal incidence ellipsometry, also known as Reflection Anisotropy Spectroscopy [4]. The observed plasmon resonances are the result of anisotropy in the local length scale. This system allows to probe quantitatively the adequacy of the RR. One of the limits discussed is the inability to discriminate between roughness and roughness length scale for small scale roughness. The ability to monitor in-situ the evolution of the anisotropy of the roughness distribution will be addressed.

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10:20am **EL+AS+EM+MS+TF-FrM7 Spectroscopic Ellipsometry on Graphene**, ***J.W. Weber***, Eindhoven University of Technology, Netherlands, *V.E. Calado,* Delft University of Technology, Netherlands, *M.C.M. van de Sanden*, Eindhoven University of Technology, Netherlands

We show how we used spectroscopic ellipsometry to determine both the optical constants and thickness of graphene1. We scanned a mechanically exfoliated graphene flake (150 x 380 μm) on an oxidized silicon wafer (98 nm SiO2) with a spectroscopic ellipsometer with a focused spot (100 x 55 μm) at an angle of 55°, in the range 210-1000 nm. The spectroscopic ellipsometric data were analyzed with an optical model in which the optical constants of graphene were parameterized by B-splines.2,3 This parameterization was key in the uncorrelated, accurate and simultaneous determination of the optical constants and thickness of graphene. The thickness is in perfect agreement with the thickness as expected from the interlayer spacing in graphite: 3.4 Å. This work opens up the possibility for in situ monitoring of graphene growth.

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10:40am **EL+AS+EM+MS+TF-FrM8 Free-charge Carrier Properties of Epitaxial Graphene by Terahertz and Infrared Ellipsometry**, ***T. Hofmann***, *A. Boosalis, P. Kühne,* University of Nebraska-Lincoln, *J.L. Tedesco, R.L. Myers-Ward, P.M. Campbell, C.R. Eddy, Jr., D.K. Gaskill,* U.S. Naval Research Laboratory, *V. Shields, S. Shivaraman, M.G. Spencer, W.J. Schaff,* Cornell University, *M. Schubert*, University of Nebraska-Lincoln

Graphene systems exhibit extremely high charge carrier mobilities highly suitable for the design of high speed terahertz (THz), Angstrom scale transistors [1]. The use of graphene for future electronic devices requires the growth of epitaxial graphene layers on suitable substrates.

We have grown highest-quality epitaxial graphene on Si- and C-faces of silicon carbide substrates under various conditions. We report on Terahertz (THz), Far-infrared (FIR) and Infrared (IR) ellipsometry and THz-IR Optical Hall-effect (generalized ellipsometry in magnetic fields) investigations of the free-charge carrier properties in epitaxial graphene samples. Furthermore, new developments on the tunable-wavelength frequency-domain THz ellipsometry instrumentation with and without external magnetic will be described [2,3].

Our ellipsometric data allows the identification of multiple, parallel sheet carrier densities within the single-to-few monolayer thick graphene layers, and which crucially depend on substrate orientation and growth condition. Analysis of the multiple two-dimensional carrier sheet densities reveals their extreme yet strongly varying mobility, effective mass, and density parameters as well as the vertical carrier sheet profile. Our findings reveal striking influences of the substrate. We discuss the physical mechanisms of the substrate that influence the free-charge carrier properties in epitaxial graphene such as surface polarity, dopant incorporation, surface roughness, and defects. We present a free-charge carrier model for epitaxial graphene, its implications due to the substrate, and discuss in light of previous implementations [4].

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11:00am **EL+AS+EM+MS+TF-FrM9 Mueller-Matrix Studies of Scarab Beetles using Spectroscopic Ellipsometry and Imaging Polarimetry**, ***H. Arwin***, Linköping University, Sweden, *S. Manhas,* LPICM, CNRS, Ecole Polytechnique, France, *J. Landin, K. Järrendahl,* Linköping University, Sweden, *A. De Martino*, LPICM, CNRS, Ecole Polytechnique, France

Many scarab beetles exhibit astonishing structural colors and polarization phenomena including circular polarization in the reflected light. Electron microscopy studies reveal that the cuticles of these beetles contain complex layered and/or chiral structures. From an applications point of view it is of large interest to explore these nanostructures as inspiration for biomimetic design based on structural colors and/or polarization.

In this work, spectroscopic ellipsometry as well as imaging polarimetry are used to determine normalized Mueller matrices of cuticles of several scarab beetle species. Mueller-matrix data in the visible spectral range are measured with a dual-rotating compensator ellipsometer. In addition real-space as well as Fourier-space Mueller-matrix images are recorded at 532 nm and 632 nm with an imaging polarimeter utilizing a microscope objective with a high numeric aperture.

The M41 Mueller-matrix element measured on the partly blue and partly green *Coptomia laevis* is very small and represents a beetle with a nanostructure without chirality. *Anoplognathus frenchi* is goldish and has a broad-band reflection with large values on M41. Very pronounced left-handed polarization effects are observed. *Cetonia aurata* has a narrow-band reflection and the color varies from green to red among species. In the color range of reflection, strong left-handed polarization is observed. Some beetles are also found to reflect right-handed polarization at certain wavelengths and angles of incidences.

The real-space Mueller-matrix images carry information about lateral variation of Mueller-matrix elements. In many cases beetle surfaces are laterally inhomogeneous. On the other hand, Fourier space images provide the azimuthal and angle of incidence variations of the Mueller-matrix elements, averaged over the spatial inhomogeneities. The Mueller-matrix

images are found to be consistent with the spectroscopic Mueller-matrix results and provide complementary information. The spectral Mueller-matrix data are also used for modelling the nanostructure of beetle cuticles involving dielectric surface layers and heliocoidal structures. Parameterization in terms of layer thicknesses, refractive indices and pitch of the helix in cuticle structures are presented.

11:20am **EL+AS+EM+MS+TF-FrM10 Agent-Free Bio-Chemical Sensing with Sculptured Thin Films**, ***D. Schmidt***, *K.B. Rodenhausen, S. Schöche, T. Hofmann, E.B. Schubert, M. Schubert*, University of Nebraska-Lincoln

A new concept of agent-free highly sensitive bio-chemical sensing is presented based on the change of birefringence of sculptured thin films (STFs). A mere intermixture of liquid or gaseous molecules with solid state STFs dramatically changes the overall optical properties of the porous film, which can be easily detected by means of ellipsometry or even the bare eye, i.e. light intensity changes upon interaction.

Previously, we have shown that STF exhibit highly anisotropic optical properties, which differ significantly from their bulk properties. Strong form birefringence and large dichroism can be tailored by appropriate deposition geometries to produce desired optical responses. In order to vary structure spacing (void fraction), and enable growth on defined sites self-assembled large-scale substrate patterning by diblock copolymer nanolithography can be exploited. We have demonstrated that generalized spectroscopic ellipsometry is ideally suited for determining geometrical structure and the anisotropic optical properties of STFs [1-3]. Recently, we have demonstrated that the combination of spectroscopic ellipsometry (SE) with quartz crystal microbalance (QCM) methods provides access to organic thin film thickness and porosity information and can further reveal structural properties of thin organic films [4].

Here we report on the application of STFs for bio-chemical sensing by simultaneous in-situ generalized ellipsometry (GE) and QCM measurements. STFs from different materials were deposited by glancing angle electron beam evaporation on gold-coated quartz crystals. Changes in the resonance frequency of the quartz crystals before and after STF deposition were used to determine the void fraction of the STF and agree well with scanning electron micrograph estimates.

In our in-situ experiments, we observe that the optical response of STFs is very sensitive to the ambient medium. We find that the filling of the STFs void fraction with solvents of different refractive index causes changes in birefringence as well as dichroism of the STFs.

Furthermore, the deposition of cetyltrimethylammonium bromide (CTAB), a well-known detergent molecule, is used here in order to demonstrate the detection of organic molecules. We find that the intriguing optical response of the STFs is changing drastically with the deposition of minute amounts of CTAB.

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