

# Thursday Morning, November 3, 2011

## Spectroscopic Ellipsometry Focus Topic

Room: 209 - Session EL+AS+EM+MS+PS+TF-ThM

## Spectroscopic Ellipsometry of Biological Materials and Organic Films

**Moderator:** M. Creatore, Eindhoven University of Technology, the Netherlands, K.G. Lloyd, DuPont Corporate Center for Analytical Sciences

8:00am **EL+AS+EM+MS+PS+TF-ThM1 Application of Various Spectroscopic Ellipsometry Techniques for In Situ Studies of Thin Polymer Films on Solid Substrates, K.-J. Eichhorn,** Leibniz-Institut für Polymerforschung Dresden e.V., Germany **INVITED**

Thin films of functional polymers are not only widely integrated into modern micro- and nanoelectronic devices, but also used to modify solid surfaces for biosensor, biomedical and antifouling applications.

Therefore, in the first part I will report on improved temperature-dependent spectroscopic Vis-ellipsometry experiments to study confinement effects on the polymer dynamics in thin films to address a question which is controversially discussed in the polymer physics community for a long time past: Is there really a significant change of the glass transition temperature  $T_g$  of polymers when confined in nanoscopic films (studied here down to about 10 nm thickness)? We compared well-known linear polystyrenes of different molecular weights /1/ and hyperbranched polyesters having different architectures and functional groups /2/. Films of different thickness were prepared by spin-coating on silicon wafers, carefully equilibrated and measured in dependence on temperature (up to 250°C) in an inert gas atmosphere. The  $T_g$  data were determined from the ellipsometric results and discussed.

In the second part, "smart" surfaces will be presented which can be used for controlled adsorption and release of biomolecules. For that, different types of stimuli-responsive polymer brushes were prepared on solid substrates by a "grafting-to" procedure. The brush properties (e.g. swelling/collapsing) as well as the resulting adsorption/desorption of model proteins (e.g. Human Serum Albumin, Chymotrypsin) can be switched in an appropriate aqueous medium with temperature (PNIPAAm) and/or pH (PAA-P2VP). The corresponding processes at the solid-liquid interface were studied in-situ by spectroscopic Vis- and IR-ellipsometry /3,4/.

/1/ M. Tress, M. Erber, E.U. Mapesa, H. Huth, J. Müller, A. Sergei, C. Schick, K.-J. Eichhorn, B. Voit, F. Kremer, *Macromolecules* 43 (2010), 9937-9944

/2/ M. Erber, A. Khalyavina, K.-J. Eichhorn, B. Voit, *Polymer* 51 (2010), 129-135

/3/ E. Bittrich, M. Kuntzsch, K.-J. Eichhorn, P. Uhlmann, *J. Polym. Sci. B, Polym. Phys.* 48 (2010), 1606-1615

/4/ Y. Mikhailova, L. Ionov, J. Rappich, M. Gensch, N. Esser, S. Minko, K.-J. Eichhorn, M. Stamm, K. Hinrichs, *Anal. Chem.* 79 (2007) 20, 7676-7682

8:40am **EL+AS+EM+MS+PS+TF-ThM3 Hard Matter Meets Thin Polymer Films-Spectroscopic Ellipsometry as a Versatile Tool to Investigate Properties of Responsive Poly(N-isopropylacrylamide) Systems with Incorporated Magnetic Nanoparticles, S. Rauch,** Leibniz-Institut für Polymerforschung Dresden e. V., Germany

Responsive polymer systems designed by using polymer brushes or hydrogels are interesting systems, which can exhibit reversible or irreversible changes in their physical and structural properties to special environmental conditions (e.g. temperature or magnetic fields). The temperature responsive poly(*N*-isopropylacrylamide) (PNIPAAm) is one of such polymers. It undergoes a phase transition in aqueous solution at its lower critical solution temperature (LCST) of 32 °C which induces an increase in hydrophobicity. Combined with the properties of magnetic nanoparticles (NP) (e.g.  $Fe_3O_4$  or  $CoFe_2O_4$ ) these systems can lead to new surface functionalities with new interesting properties for many applications, as sensing, wettability or (bio)adhesion.

For the design of such thin film systems a basic knowledge of the film characteristics is essential. Therefore it is first necessary to know how much nanoparticles are inside or attached to the system. By using spectroscopic Vis-Ellipsometry (SE) it is not only possible to investigate optical properties of these films but also the composition of it, e.g. volume fraction of  $Fe_3O_4$ -NP.

We studied two types of films and present results for a thin NP-composite film prepared by pre-mixing of the PNIPAAm with hydrophobic  $Fe_3O_4$ -NP, spin-coated and grafted to a silicon substrate (System 1) and a film prepared by adsorption of hydrophilic functionalized  $Fe_3O_4$ -NP onto PNIPAAm brushes (System 2). The former system was chosen to develop an optical model starting from a simple two component effective-medium-approach (Maxwell-Garnett-EMA) using the optical constants of the pure polymer measured by SE and of  $Fe_3O_4$  with averaged data taken from three different publications. The SE best fit-results were validated against scanning electron microscopy (SEM), atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS).

After transferring this optical model to System 2, the adsorption of hydrophilic functionalized  $Fe_3O_4$ -NP onto PNIPAAm brushes was investigated and will be discussed with additional results obtained from contact angle (CA) and phase transition (LCST) measurements.

9:00am **EL+AS+EM+MS+PS+TF-ThM4 The White Scarab Beetle *Cyphochilus insulanus* -Scattering and Polarization Properties, C. Akerlind,** Swedish Defence Research Agency / Linköping University, Sweden, *H. Arwin,* Linköping University, Sweden, *T. Hallberg, H. Kariis,* Swedish Defence Research Agency, Sweden, *J. Landin, K. Järrendahl,* Linköping University, Sweden

Three methods were used to characterize the optical properties of the light scattering white scarab beetle *Cyphochilus insulanus*. Spectral directional hemispherical (DH) reflectance measurements in the wavelength region 250 nm to 25  $\mu$ m were performed using integrating spheres. The general spectral appearance shows a relatively strong reflectance band in the range 400 - 1600 nm. The Bidirectional Reflection Distribution Function (BRDF) was measured over a semi-circle in the plane of incidence at the wavelengths 633 nm and 3.39  $\mu$ m, using s- and p-polarized light. In the visible the BRDF data shows a near Lambertian behaviour with a constant BRDF for most angles, i.e. the light is diffusely scattered. For large incident angles and in the infrared the BRDF is more specular. Full Mueller-matrix spectroscopic ellipsometry (MMSE) measurements were performed in the wavelength range 250 - 1000 nm at angles of incidence between 45° and 75° using a dual rotating compensator ellipsometer. The Mueller data show that the reflected light in general has a high degree of polarization  $P$  even for nonpolarized incident light. The angular dependence of  $P$  was pronounced for incident p-polarized light and was very low near the Brewster angle. In comparison,  $P$  was high for all measured angles for incident s-polarized light. Close to the Brewster angle, the reflected light showed a high ellipticity for incident p-polarized light. The optical data is used to model the structure and optical response of the beetle cuticle. With input from the DH and BRDF-data analysis of the Mueller Matrix data was made using Fresnel-based layer modelling. 2-, 3- and n-phase models are compared. The obtained optical properties are also used to calculate color data in terms of chromaticity and whiteness. The scattering properties of the *Cyphochilus insulanus* cuticle are also discussed by combining the BRDF and MMSE data as well as recently obtained Mueller matrix imaging polarimetry results.

9:20am **EL+AS+EM+MS+PS+TF-ThM5 Real-time Spectroscopic Ellipsometry and Quartz Crystal Microbalance with Dissipation Characterization of Biomolecule Adsorption within Sculptured Thin Films, T. Kasputis, D. Schmidt, K.B. Rodenhause, H. Wang, A.K. Pannier, M. Schubert,** University of Nebraska - Lincoln

Recent improvements in nanofabrication strategies have led to the development of precisely crafted nanostructures with intricate features. Incorporating biomolecules such as proteins, DNA, drugs, and even whole cells could allow for functionalization of nanostructured surfaces for biological applications including biosensing, tissue engineering scaffolds, and drug and gene delivery. Along with the nanofabrication of biological devices, there is a need to develop instrumentation capable of probing and characterizing the dynamic evolution of these bio-functionalized interfaces. Spectroscopic ellipsometry combined with quartz crystal microbalance with dissipation (SE/QCM-D) is a non-destructive optical/mechanical characterization technique that reveals dynamic properties, including average film thickness (with sub-angstrom resolution), adsorbed mass, and porosity.

Nanostructures in the form of sculptured thin films (STF) were fabricated by glancing angle deposition via electron beam evaporation of titanium onto gold-coated quartz sensors. The sensors were then mounted within an SE/QCM-D liquid cell, and proteins of varying sizes were deposited and characterized, *in-situ*. Protein adsorption was detected shortly after introducing the protein solutions by SE and QCM-D as a change in the optical response and decrease of vibration frequency, respectively. QCM-D reported greater adsorbed mass for larger proteins (fibronectin) than smaller

proteins (bovine serum albumin). The adsorbed mass of proteins within the nanostructured scaffold exceeded that of proteins on flat surfaces, confirming that the STFs are capable of trapping proteins. Analysis of the anisotropic optical response from the nanostructures, which is very sensitive to environmental changes, adds complementary information on protein adsorption; the optical quantification is in agreement with QCM-D results. In addition, the adsorption of other biomolecules, such as cells and DNA complexes, has also been accomplished. The use of combinatorial SE/QCM-D to characterize and monitor the attachment of biomolecules on complex nanotopographies will improve the design and fabrication strategies for a wide array of biotechnological devices.

9:40am **EL+AS+EM+MS+PS+TF-ThM6 Characterization of Multilayer Organic Thin Film for Use as an Aptamer Biosensor with Hybrid Spectroscopic Ellipsometry and Quartz Crystal Microbalance with Dissipation**, *J.Y. Gerasimov, K.B. Rodenhausen, H. Wang, R.Y. Lai, M. Schubert*, University of Nebraska - Lincoln

DNA aptamer molecules passivated by alkanethiols can be used for biological detection and screening. Through the use of spectroscopic ellipsometry (SE, optical) and quartz crystal microbalance with dissipation (QCM-D, mechanical) techniques, selective binding of analytes to chemisorbed aptamer probes can be observed *in-situ*. The system analyzed consists of a gold-coated quartz substrate, a multilayer organic thin film (containing aptamer probe, alkanethiol, and single-stranded DNA analyte), and physiological buffer solution. The attachment and detachment of material, the hybridization efficiency of the aptamer probes, and changes in the porosity of the multilayer organic thin film were all determined by SE/QCM-D.

In this contribution, we present the real-time SE/QCM-D characterization of (a) the formation of the aptamer probe layer, (b) the subsequent chemisorption of alkanethiol, and (c) the interrogation of single-stranded DNA that is non-complementary or complementary to the sequence found on the aptamer probe. The aptamer DNA sequence encodes codon 12 of the *K-ras* gene; mutations of this gene are frequently found among pancreatic cancer patients. We found that introduction of either complementary or non-complementary DNA caused increases of the multilayer organic thin film thickness. However, our SE/QCM-D analysis showed that the porosity of the multilayer organic thin film responded differently depending on the compatibility of the DNA analyte. The SE/QCM-D technique provides evidence for different surface attachment mechanisms and can be useful in characterizing biological interfaces.

10:40am **EL+AS+EM+MS+PS+TF-ThM9 Contamination Processes of EUV Optics Characterized by Spectroscopic Ellipsometry**, *L.J. Richter, C. Tarrío, S. Grantham, S.B. Hill, T.B. Lucatorto*, National Institute of Standards and Technology, *N.S. Faradzhev*, University of Virginia

Extreme ultraviolet (EUV) lithography using 13.5 nm light is emerging as a viable tool for semiconductor fabrication at design rules below 32 nm. Tool performance critically depends on limiting and mitigating degradation of the EUV optical elements. A primary degradation mechanism is the EUV induced deposition of carbon from ambient species originating from outgassing of the unbaked vacuum system and/or outgassing from the EUV irradiation of the resist. Using the high brightness of the Synchrotron Ultraviolet Radiation Facility (SURF-III) at NIST both fundamental studies of EUV induced contamination and practical ("witness plate") studies of resist out gassing are performed. Typical deposits are hydrogenated amorphous carbon features with peak thicknesses of about 1 nm and nominal lateral extent of 1  $\mu$ m. Both ex-situ, small spot mapping spectroscopic ellipsometry (SE) and in situ single-wavelength imaging nulling-ellipsometry are used for rapid, sensitive, contamination metrology. Fundamental studies of contamination by admitted gases indicate that the process is a complex function of both gas pressure and photon dose. Correlations between SE and XPS are suggestive that, at high EUV fluxes, densification can occur resulting in spatial variation in the deposit dielectric function. The use of principal component analysis of the SE images to highlight the spatial diversity will be discussed.

11:00am **EL+AS+EM+MS+PS+TF-ThM10 Characterization of Organic Solar Cells Materials and Structures by Spectroscopic Ellipsometry**, *J.P. Piel, L. Kitzinger, A. Bondaz, C. Defranoux*, SEMILAB-SOPRALAB, France

Spectroscopic Ellipsometry (S.E) is a well known optical technique widely used for the characterisation of all types of thin films for determination of film thickness and optical indices on glass or plastic substrates.

S.E. is also being applied to the characterization of materials and multilayer structures of organic materials like organic light-emitting diodes (OLEDs) or Organic Solar Cells.

We present the determination of the refractive indices of organic Solar Cells materials like P3HT, PCBM, Pentacene, Perylene, and their blends. Complex organic materials can be analyzed accurately and fully characterized from their absorption bands in the visible and UV range (190nm to 900nm).

Transmission and absorption can be also measured at the same time and be used to determine the optical properties of these materials.

Using these refractive indices, analysis of real multi-layer stacks can be done. The refractive indices can be used afterwards to automatically optimise and balance the energy flow dissipation  $Q$  inside an organic solar cell composed of a thin film stack. We present an example performed on a single cell and on a tandem bi-layer cell structure.

Since these materials are sensitive to moisture and pollution, it can be necessary to measure their optical properties and thickness values through an encapsulated media. We will demonstrate how we can measure single layer properties and multi-layer stacks, through encapsulated samples, from the back side of the substrate. This technique can be applied to test structure or real Organic Solar Cell monitoring. Backside measurement combined with a water vapour cell used for ellipsometry porosimetry is used to test the efficiency of the thin film encapsulation.

We also present the characterization of ITO and ZnO transparent electrodes by S.E. and how near infra-red ellipsometry is used to determine the ITO resistance, without contact, by using the Drude behaviour on encapsulated samples.

# Thursday Afternoon, November 3, 2011

## Spectroscopic Ellipsometry Focus Topic

Room: 209 - Session EL+AS+EM+MS+PS+TF-ThA

### Spectroscopic Ellipsometry for Photovoltaics, Metals and Oxide Thin Films

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

#### 2:00pm EL+AS+EM+MS+PS+TF-ThA1 Applications of Ellipsometry in Photovoltaics, D. Levi, National Renewable Energy Laboratory INVITED

With the growing possibility of anthropomorphic-induced climate change there has come increasing concern over energy-related emissions of carbon dioxide into the atmosphere. The search for low or no-carbon energy sources has intensified. This has led to a twenty first century gold rush into photovoltaics research and technology startups. Although the PV industry has maintained its exponential growth rate through the global economic downturn, electricity from photovoltaics is still a long ways from economic competitiveness with fossil fuel-based electricity sources. The U.S. Department of Energy recently announced the Sunshot program, with the expressed goal of \$1/Watt installed cost for utility scale PV plants by 2017. This aggressive goal will require radical advances in new and existing PV technologies.

This presentation will begin with an overview of the major PV technologies and the state of the rapidly evolving global photovoltaics industry. Photovoltaics is a natural arena for application of spectroscopic ellipsometry. Nearly all PV devices are made of multiple thin films of semiconductors and transparent conducting oxides. New materials are constantly being introduced. Film thickness, optical properties, interfaces, electronic properties, and film growth dynamics are all critical aspects of these devices and lend themselves to investigation through the use of spectroscopic ellipsometry. I will present several case studies of how we have applied spectroscopic ellipsometry in our research in photovoltaics at the National Renewable Energy Laboratory.

#### 2:40pm EL+AS+EM+MS+PS+TF-ThA3 Comparison between Ex Situ and Real Time Spectroscopic Ellipsometry Measurements of Structurally Graded Si:H Thin Films, N.J. Podraza, University of Toledo

Analysis of spectroscopic ellipsometry measurements of graded thin films remains challenging, although analysis procedures and software have improved over the past several decades. Practical use of these processes remains somewhat time consuming and is often not fully utilized by the casual user. In this work, ex situ ellipsometric spectra collected for static samples and real time spectroscopic ellipsometry (RTSE) measurements collected during film growth will be compared to illustrate differences in results arising from the measurement procedures and analysis. As an application, consider hydrogenated silicon (Si:H) thin films used for solar cells. Devices typically incorporate either amorphous silicon (a-Si:H) or "nanocrystalline" silicon (nc-Si:H) absorber layers, although the best "nanocrystalline" absorber layers actually consist of mixed-phase amorphous+nanocrystalline (a+nc) material. Si:H thin films may initially (i) nucleate as amorphous and remain amorphous throughout growth; (ii) immediately nucleate as nanocrystallites; or (iii) initially evolve in the amorphous regime but nucleate crystallites which subsequently grow preferentially over the surrounding amorphous material until nanocrystallite coalescence. Analysis of ellipsometric spectra collected for (i) or (ii) simply involve using a substrate / bulk film / surface roughness model and complex dielectric function spectra ( $\epsilon = \epsilon_1 + i\epsilon_2$ ) for the bulk material. For (iii), RTSE is ideally used to monitor the growth of Si:H that evolves through the amorphous, nanocrystalline, and mixed-phase regimes and a virtual interface analysis (VIA) procedure is used to extract  $\epsilon$  for the amorphous and nanocrystalline components, the bulk and surface roughness thicknesses versus time, and the nanocrystalline fraction depth profile in the (a+nc) growth regime. For (a+nc)-Si:H films only measured with a single static ex situ measurement at the end of the deposition, obtaining  $\epsilon$  and structural parameters of the film become less precise. Specifically, sensitivity to the variation in the nanocrystallite fraction with thickness may be lost and inaccurate  $\epsilon$  for the component materials may be obtained. This work seeks to compare the structural and optical properties of (a+nc)-Si:H obtained by RTSE and VIA with those from analysis of static ex situ spectra with models using different structures, parameterizations in  $\epsilon$ , and spectral range restrictions. These comparisons will be used to identify appropriate structural and dielectric function models to more accurately analyze

structurally graded thin films under different material and measurement circumstances.

#### 3:00pm EL+AS+EM+MS+PS+TF-ThA4 Real-Time Spectroscopic Ellipsometry of Cu(In,Ga)Se<sub>2</sub> Thin Film Deposition: Copper Transition in 3-Stage Co-Evaporation Process, D. Attygalle, University of Toledo, V. Ranjan, Old Dominion University, P. Aryal, University of Toledo, S. Marsillac, Old Dominion University, R.W. Collins, University of Toledo

With record efficiencies above 20%, Cu(In,Ga)Se<sub>2</sub> (CIGS) based solar cells have shown the greatest potential for success among the thin film photovoltaics technologies. Thermal co-evaporation of individual elements has proven to produce extremely high quality CIGS materials, provides a high level of flexibility, but also generates greater challenges in process optimization. The limitations of existing process monitoring capabilities, hence the challenge of correcting process fluctuations in real time, has led the industrial community toward more controllable CIGS deposition processes. Real time spectroscopic ellipsometry (RTSE) can be used successfully in the monitoring of complicated processes -- including CIGS film preparation by co-evaporation using precursor films of (In<sub>x</sub>Ga<sub>1-x</sub>)<sub>2</sub>Se<sub>3</sub>. Information extracted from RTSE includes the evolution of bulk layer and surface roughness layer thicknesses, the composition and phase, as well as the layer dielectric functions, all of which can assist in understanding the fabrication process and in optimizing solar cells. In this study, the focus is on the transitions of Cu-poor to Cu-rich CIGS and vice versa by observing the changes in ( $\psi$ ,  $\Delta$ ) spectra obtained by RTSE. The commonly used monitoring method, which involves observing the changes in emissivity of the film, largely depends on the apparatus design, the substrate, and the bulk layer thickness. When a CIGS film is prepared by exposing a precursor film of (In<sub>x</sub>Ga<sub>1-x</sub>)<sub>2</sub>Se<sub>3</sub> to Cu and Se fluxes, thereby becoming Cu-rich, a semi-liquid Cu<sub>2-x</sub>Se phase is believed to form on top of a bulk layer consisting of mixed phases of Cu(In,Ga)Se<sub>2</sub> and Cu<sub>2-x</sub>Se [1]. A multilayer optical model, with appropriate effective medium approximation layers to represent this scenario, has shown good agreement with the observed ( $\psi$ ,  $\Delta$ ) spectra. Since RTSE is highly sensitive to monolayer-level changes in the top-most layer, RTSE gives superior sensitivity in Cu-rich to Cu-poor end point detection, which occurs when the top Cu<sub>2-x</sub>Se phase drops below detectable limits. Furthermore this method is less affected by the substrate and bulk layer thickness. Although careful analysis of RTSE can give a wealth of information about CIGS material properties and their evolution, this type of end point detection can be successful simply by monitoring the real time changes in the ( $\psi$ ,  $\Delta$ ) spectra.

[1] J. AbuShama, R. Noufi, Y. Yan, K. Jones, B. Keyes, P. Dippo, M. Romero, M. Al-Jassim, J. Alleman, and D.L. Williamson, "Cu(In,Ga)Se<sub>2</sub> Thin-film evolution during growth from (In,Ga)Se<sub>3</sub> precursors", *Mat. Res. Soc. Symp. Proc.* paper H7.2.1, (2001).

#### 3:40pm EL+AS+EM+MS+PS+TF-ThA6 Bulk Heterojunction Solar Cell Characterization by Phase Modulated Spectroscopic Ellipsometry, K. Uppreddi, L. Yan, HORIBA Scientific

The blend morphology, phase separation as well as crystallinity of organic photovoltaic solar cell are important properties to increase the efficiency. The performance of such cells is strongly influenced by blend composition and thermal annealing conditions. In this work we demonstrate the use of ellipsometry as a powerful and sensitive metrology means of monitoring organic solar cell based on the blend of poly(3-hexylthiophene) (P3HT) and [6,6]-phenyl C61-butyric acid methyl ester (PCBM). Ellipsometric measurements were performed on P3HT/c-Si, PCBM/c-Si and P3HT:PCBM/c-Si at an angle of incidence of 70 degree, across the spectral range 190 – 2100 nm (0.6-6.5 eV). Two different analysis protocols were used to model the P3HT:PCBM blend structure. In the first protocol effective medium theory was used to represent the optical constant of layer, where as in the second one the blend was treated as one single homogenous material. The approach renders investigation of final morphology and composition.

#### 4:00pm EL+AS+EM+MS+PS+TF-ThA7 In Situ Spectroscopic Ellipsometry during Atomic Layer Deposition of Pt, Pd and Ru, N. Leick, J.W. Weber, M.J. Weber, A.J.M. Mackus, H.C.M. Knoops, W.M.M. Kessels, Eindhoven University of Technology, Netherlands

The precise thickness control of atomic layer deposition (ALD) and its conformal growth make ALD the method of choice for nanometer thin film deposition. Platinum-group metals such as Pt, Pd and Ru have many applications in the areas of nanoelectronics and catalysis and recently there has been considerable interest to deposit films of these materials by ALD. Spectroscopic ellipsometry (SE) is a powerful, noninvasive optical technique that can be used *in situ* during ALD to precisely monitor the thickness of the films. SE also provides information on the optical and

electrical properties of the films which is very relevant for their applications. Choi et al. [1] previously investigated the dielectric functions of Pt-group metal films with a thickness of ~400 nm as prepared by physical vapor deposition. For the aforementioned applications, however, the films are required to be much thinner, which leads to differences in film morphology as well as to dielectric functions that can be different from those of bulk films. In the spectroscopic ellipsometry work to be presented in this contribution we have therefore focused on films with thicknesses from 5 nm to 35 nm. *In situ* data was obtained during ALD in the photon energy range of 0.7 – 6.5 eV. Using a Kramers-Kronig consistent B-spline model to account for the thickness-dependent dielectric functions, we were able to obtain accurate ALD growth-per-cycle values for Ru, Pt and Pd ( $1.00 \pm 0.06 \text{ \AA}$ ,  $0.47 \pm 0.04 \text{ \AA}$ ,  $0.14 \pm 0.02 \text{ \AA}$ ). Furthermore, the contributions from free-carriers (Drude term) and interband absorptions (Lorentz-oscillator contributions) were investigated by combining the SE data with FT-IR reflectance data such that the photon energy range of 0.4 eV – 6.5 eV was covered. In this range, it was possible to represent each film with a unique Drude-Lorentz model although some ambiguities about the Lorentz oscillator contributions remained in the case of Ru. It will be shown that the extracted thicknesses and electrical resistivities from this model are in line with data obtained from X-ray reflectometry and four-point probe measurements (for example Ru:  $\rho_{SE} \sim 23 \mu\Omega\cdot\text{cm}$  and  $\rho_{FPP} \sim 16 \mu\Omega\cdot\text{cm}$ ). Furthermore, in the case of Ru also the influence of the film roughness will be addressed.

[1] Choi et al., Phys. Rev. B **74**, 205117 (2006)

4:20pm **EL+AS+EM+MS+PS+TF-ThA8 Manipulating the Optical Properties of Metals: Sculptured Thin Films Coated by Atomic Layer Deposition**, D. Schmidt, N. Ianno, E. Schubert, M. Schubert, University of Nebraska - Lincoln

The fabrication of three-dimensional metal nanostructures with tailored geometry is one of the central challenges of nanotechnology because geometrical and material parameters are responsible for the optical, electrical, mechanical, chemical, or magnetic properties of such nanostructured thin films. Engineered artificial sculptured thin films (STFs) with designed anisotropies are potential candidates for applications in various fields such as optics, magneto-optics, as well as chemical and biological sensing and detection. However, in order to utilize metallic nanostructures for novel applications their size-, structure-, and material-driven physical properties have to be understood and quantified.

We utilize glancing angle electron-beam deposition, which exploits physical atomic-scale shadowing and dynamically varying particle flux azimuth for fabrication of three-dimensional highly spatially coherent STFs with different morphologies. Subsequently, nanostructures are individually covered with a thin conformal coating (cladding) by means of atomic layer deposition (ALD).

We will present the anisotropic optical properties of highly anisotropic ALD coated metal STFs determined by generalized spectroscopic ellipsometry in the visible and near-infrared spectral region. The analysis of our multilayer slanted columnar thin films deposited at glancing angle ( $\theta_i = 85^\circ$ ) revealed that such STFs possess monoclinic optical properties, and the optical response may be described by an effective medium dielectric homogenization approach. It will be discussed how the anisotropic Bruggeman effective medium approximation (AB-EMA) allows for determination of structural parameters as well as fractions of individual film constituents. Furthermore, the AB-EMA analysis reveals that the anisotropic dielectric properties of the metal core changes upon deposition of a dielectric cladding.

4:40pm **EL+AS+EM+MS+PS+TF-ThA9 Ellipsometric Characterisation of Porous Aluminium Oxide Supports**, W. Ogieglo, N.E. Benes, H. Wormeester, MESA+ Institute for Nanotechnology, University of Twente, Enschede, The Netherlands

Porous aluminium oxide is widely used as a support material for thin film inorganic micro- and mesoporous membranes. Such membranes are used in energy-efficient gas separation, pervaporation and nanofiltration processes. Ellipsometry can be used to determine material properties of the thin membrane films, as well as the penetrant loading [1]. Interpretation of the ellipsometry data requires a detailed knowledge of the porous aluminium oxide support. This support is made of aluminium oxide particles that are sintered together. In between the particles voids are present that amount to 38% porosity. We have studied the influence of the size of the voids on the optical response of the support material. For this study, voids with a diameter of around 60, 80 and 160 nm were used. We noted a strong decrease of the normal incidence specular reflection with void size and a subsequent increase in off specular reflection. In ellipsometry, only a limited depolarization of the specular reflected light was noted in the wavelength range between 300 and 1750 nm. The angle dependent ellipsometry measurements showed that the optical properties of these

supports can not be obtained from a direct inversion. The reason for this is that at the interface the more or less spherical voids are cut, which leads to a distribution of openings at the surface, i.e., a substrate with a very rough surface. This roughness was modelled with a graded porosity changing from 38% in the bulk to 75% at the outer surface. This measured variation in porosity is very similar to the cumulative height distribution of the surface layer obtained from AFM. The validity of this graded porosity model was verified from the analysis of a sample with a thin polysulfone (PSU) layer deposited on the support. The PSU layer partly fills the open pores at the surface. This results in an interface with a graded variation in aluminium oxide, void and PSU.

The proper treatment of the surface layer also provides the optical properties of the porous aluminium oxide bulk material itself. These optical properties can in a limited wavelength range be modelled with Bruggeman's effective medium approximation. As a consequence of the size of the inclusions, their diameter is no longer negligible with respect to the wavelength of light in the UV part of the spectrum. For the material with the largest pore size, also a large part of the visible range has to be excluded. A more elaborate approach than the standard effective medium approach has to be used in this case.

[1] H. Wormeester, N.E. Benes, G.I. Spijksma, H. Verweij and B. Poelsema Thin Solid Films **455-456**, 747-751 (2004)

5:00pm **EL+AS+EM+MS+PS+TF-ThA10 Optical Properties and Structure of Vanadium Oxide Thin Films**, M.A. Motyka, M.W. Horn, Pennsylvania State University, N.J. Podraza, University of Toledo

Vanadium oxide ( $\text{VO}_x$ ) thin films are common materials used as imaging layers in uncooled microbolometer based thermal imaging devices. These films are used in this application largely due to the controllable resistivity of the film ( $\rho$ ), the high temperature coefficient of resistance (TCR), and the low electrical noise. One of the main difficulties of this material system relates to the multiple valence states of vanadium, each of which results in materials with different electrical properties. Bolometer quality  $\text{VO}_x$  may consist of a composite of nanocrystalline face centered cubic (FCC) VO phase and amorphous materials. The thin film oxygen content via Rutherford back scattering (RBS) has suggested that the typical ratio V:O should be near 1:1.7-2.0, significantly higher than the stability window of the FCC phase. This off-stoichiometry ratio suggests that the amorphous material is a mixture of higher oxygen valence states similar to  $\text{V}_2\text{O}_5$  and  $\text{VO}_2$ . The higher quality  $\text{VO}_x$  thin film material also has been observed via transmission electron microscopy (TEM) to contain  $\text{VO}/\text{V}_2\text{O}_5$  nano-twin crystalline domains. The presence of each of these phases impacts the electrical and optical properties of the resulting  $\text{VO}_x$  film. Films with various oxygen contents and structures were studied with spectroscopic ellipsometry (SE) over a spectral range of 0.05 to 5.15 eV using a multichannel dual rotating compensator near-ultraviolet to near infrared instrument in conjunction with Fourier transform infrared spectroscopic ellipsometry (FTIR-SE). Thus, the complex dielectric function spectra ( $\epsilon = \epsilon_1 + i\epsilon_2$ ) can be obtained for these materials over the full spectral range. Differences in  $\epsilon$  due to variations in the film structure are observed as functions of processing, indicating that SE is a means of probing the material composition and structure. Specifically,  $\epsilon$  are compared for various film composites fabricated by unbiased pulsed DC magnetron sputtering as well as composite films prepared by reactive ion beam sputtering and pulsed DC magnetron sputtering with a substrate bias. The microstructure and  $\epsilon$  are correlated with films exhibiting the desirable device electrical properties. *In situ* real time spectroscopic ellipsometry (RTSE) has shown that environmental conditions alter the as-deposited  $\text{VO}_x$  thin films grown via pulsed DC-magnetron reactive sputtering of a metallic vanadium target. In order to prevent undesired atmospheric effects to the thin film, it is a common practice to encapsulate the thin film with a more environmentally stable material. In this study, the material chosen was  $\text{SiO}_2$  grown in the same deposition chamber, pre-atmospheric exposure, via rf sputtering.

5:20pm **EL+AS+EM+MS+PS+TF-ThA11 Sensitivity of Dielectric Properties of Vanadium Dioxide Thin Films to Growth Conditions**, D.W. Ferrara, R.E. Marvel, J. Nag, R.F. Haglund, Vanderbilt University

Vanadium dioxide ( $\text{VO}_2$ ) is a strongly-correlated electron material with a well-known semiconductor-to-metal transition (SMT) that can be induced thermally ( $T_c = 68^\circ\text{C}$ ), optically, or electrically. Recently,  $\text{VO}_2$  films have attracted attention as a component in active metamaterials, especially in conjunction with metal nanostructures. Since these structures are highly sensitive to the dielectric properties of the embedding material, the SMT of  $\text{VO}_2$  can be used to tune the optical response of the structure. Accurately modeling the behavior of these structures requires detailed knowledge of the dielectric function of  $\text{VO}_2$  as it undergoes the SMT; however, previous measurements of the optical constants of  $\text{VO}_2$  reveal significant variations between experiments.

To understand systematic variations due to growth conditions, films of  $\text{VO}_2$  were deposited on either silicon, glass, or sapphire substrates by pulsed laser ablation of vanadium metal targets in 10 mTorr oxygen ( $\text{O}_2$ ) background gas, followed by annealing at  $450^\circ\text{C}$  in 250 mTorr of  $\text{O}_2$ . Anneal times were varied from 30 to 90 depending on film thickness; deposition thickness was varied from 20 nm to 200 nm. For each sample, temperature-dependent spectroscopic ellipsometry measurements at optical and near-infrared wavelengths were conducted to determine the dependence of the optical constants on film thickness, substrate and crystallinity, and temperature.

Bruggeman and Maxwell-Garnett effective-medium formulations were used to account for three constituent materials: semiconducting  $\text{VO}_2$ , metallic  $\text{VO}_2$ , and vanadium pentoxide ( $\text{V}_2\text{O}_5$ ). The effective dielectric functions were modeled using Lorentz and Tauc-Lorentz oscillators. Our results show that the contribution of  $\text{V}_2\text{O}_5$  to the effective dielectric function increases with annealing time, consistent with previous studies. The results are also substantiated using Rutherford backscattering, X-ray photoelectron spectroscopy and X-ray diffraction.

# Thursday Afternoon Poster Sessions

**Spectroscopic Ellipsometry Focus Topic**  
**Room: East Exhibit Hall - Session EL-ThP**

## Spectroscopic Ellipsometry Poster Session

**EL-ThP1 Microstructure and Dispersive Optical Parameters of Tungsten, Titanium and Tungsten-Titanium Films, V.V. Atuchin, T.I. Grigorieva, A.S. Kozhukhov, V.N. Kruchinin, L.D. Pokrovsky, Institute of Semiconductor Physics, Russian Federation, R.S. Vemuri, Pacific Northwest National Laboratory, C.V. Ramana, University of Texas at El Paso**

Tungsten (W) and titanium (Ti) films are widely used in electrochemistry, microelectronics, energy conversion and nanotechnology. In integrated optics, the nanometric Ti films are used as a source for doping LiNbO<sub>3</sub> and LiTaO<sub>3</sub> substrate and optical waveguide fabrication by thermal diffusion. Because effective refractive indices of the waveguide modes are strongly dependent on the optical profiles in doped layer, precise control of Ti film thickness ( $h$ ) is needed in the range  $h \sim 10\text{--}50$  nm. Ellipsometry can be successfully applied for nondestructive determination of the thickness of a dielectric and semi-transparent metal film when optical constants of the material are known. Regrettably, noticeable scattering was found for optical constants reported earlier in literature for W and Ti films and crystals. As it seems, this scattering appeared due to different film quality and surface state. The focus of the present work is centered on W, Ti and W-Ti film fabrication and evaluation of their optical parameters with spectroscopic ellipsometry. Tungsten and tungsten-titanium films were prepared by magnetron sputtering deposition in vacuum below 10<sup>-5</sup> Torr. Titanium films were fabricated by thermal evaporation method in vacuum below 10<sup>-5</sup> Torr. The substrate temperature was  $T=100$  °C. For precise determination of optical parameters, thick metal films ( $h \sim 100$  nm by as determined from optical interferometry) were prepared on silica substrate. To increase the metal adhesion, the substrate was subjected to RCA chemical cleaning just before insertion into vacuum chamber. Structural parameters of metal films were studied with reflection high-energy electron diffraction (RHEED). Surface micromorphology was controlled with atomic force microscopy (AFM). Spectral dependencies of refractive index  $n(\lambda)$  and extinction coefficient  $k(\lambda)$  were determined with the help of spectroscopic ellipsometry in the spectral range,  $\lambda \sim 250\text{--}1030$  nm. A relation between optical constants of pure metal W and Ti and mixed metal W-Ti films is discussed.

**EL-ThP2 Temperature Dependences of the Dielectric Response of InSb Measured by Spectroscopic Ellipsometry, J.J. Yoon, T.J. Kim, S.Y. Hwang, M.S. Diware, Y.D. Kim, Kyung Hee University, Republic of Korea, Y.C. Chang, Academia Sinica, Taiwan, Republic of China**

InSb is a promising material for optical devices, particularly for high-frequency and nonlinear-optical applications. InSb has a high electron mobility and offer excellent design flexibility as a result of its large conduction-band offset in multilayer structures. Consequently, InSb offers significant potential for devices such as quantum-well lasers, laser diodes, and heterojunction bipolar transistors. A knowledge of the dielectric function at various temperatures is required for optimizing the properties for specific device applications. In-situ control of growth is also becoming an important technique. Therefore, the dielectric function at growth temperatures is also needed. On the other hand, critical point (CP) energies can be better identified from low-temperature data, where the decreased electron-phonon interaction allows separation of CP structures that are nearly degenerate at room temperature.

Although the optical properties of InSb have been well studied, there are only a few reports of their temperature dependence in the 1.2 to 5.6 eV spectral ranges [1]. Here, we report results of an investigation of the temperature dependence of the dielectric response of InSb from 22 K to 700 K and from 0.74 to 6.57 eV.

Spectroscopic ellipsometric (SE) data were obtained on a bulk semi-insulating InSb (100) substrate. The cryostat consisted of a stainless-steel chamber with high-quality stress-free fused-quartz windows. To avoid condensation at low temperatures, the sample was maintained in ultrahigh vacuum during measurement. SE data were obtained at an angle of incidence of 70.41° using a conventional rotating-compensator system with a diode-array detector. The influence of the oxide overlayer was removed mathematically by a multilayer calculation. In the  $E_2$  energy region only four structures are clearly resolved at 300 K. However, at 22 K the  $E_2'$  and  $E_2$  structures are seen to consist of five CPs. We identified the origin of these structures with band-structure calculations using the LASTO method. Separation of the  $E_0'$ ,  $E_0'+\Delta_0'$ ,  $E_2$ ,  $E_2+\Delta_2$ ,  $E_2'$ ,  $E_1'$  and  $E_1'+\Delta_1'$  CPs was clearly found in the region of the  $E_2$  peak. Two saddle-point transitions,  $\Delta_5^{\text{cu}}\text{--}\Delta_5^{\text{vu}}$  and  $\Delta_5^{\text{cl}}\text{--}\Delta_5^{\text{vu}}$ , are clearly seen. We also determined the temperature

dependences of the newly observed transitions near 5.9 eV. These results will be useful in a number of contexts, including the design of optoelectronic devices based on InSb, as data for improved band structure calculations, and for in-situ monitoring.

[1] S. Logothetidis, L. Vina and M. Cardona, Phys. Rev. B **31**, 947 (1985).

**EL-ThP3 Tailored Helical Nanostructures Investigated with Mueller Matrix Ellipsometry, R. Magnusson, J. Birch, C.-L. Hsiao, P. Sandström, H. Arwin, K. Järrendahl, Linköping University, Sweden**

Metamaterials showing chiral features in the optical spectral range have been fabricated with the aim to obtain polarized reflection with high ellipticity.

A series of tailored anisotropic and transparent structures of helical Al<sub>1-x</sub>In<sub>x</sub>N nanorods were grown using UHV magnetron sputtering on sapphire substrates. Due to an internal in-plane composition gradient across the crystalline structure, the nanorods will tilt relative to the substrate normal. By rotating the substrate step-by-step around its normal during deposition 'staircase' helical structures are obtained. The layer thickness for each step is controlled to tailor the nanorods. Samples with different pitch and layer thickness and with right-handed as well as left-handed chirality were grown.

Ellipsometric measurements were performed using a dual rotating compensator ellipsometer providing the full Mueller matrix in the spectral range 245-1700 nm at multiple angles of incidence and 0-360° sample orientation. The relation between the optical characteristics of the samples, specifically the ellipticity, and structural parameters such as number of layers, layer thickness and nanorod pitch of the samples, was studied. For certain wavelengths, near circular polarization is observed both for right- as well as left-handed helical structures.

Based on the Mueller matrix data, descriptions of the polarization states and degree of polarization in reflection for different incoming states of polarization will be presented.

**EL-ThP4 Study of the Thin Film Growth of Volatile Condensable Material via In Situ Ellipsometry and Quartz Crystal Microbalance Measurements, J. Pu, F. Zhou, N.J. Ianno, The University of Nebraska**

DC-93-500, SCV-2590 and SCV-2590-2 silicone/siloxane based copolymer serve as adhesive components in communications satellites and other spacecraft under adverse low-earth and geo-synchronous orbits. The outgassing and deposition of Volatile Condensable Materials (VCM's) from these adhesives onto optically-sensitive surfaces of satellites is of significant interest to spacecraft-contamination engineers. In our work, samples of these materials are heated to 100 C in a liquid nitrogen (LN2) cooled cryo-shroud lined high vacuum chamber. One MK-18 quartz crystal microbalance (QCM) sensor is placed above the effusion cell which is mounted on the bottom of the chamber. At various QCM temperatures from 120K to 180K, we observed the formation of a thin film of volatile contaminant material on the gold coated QCM crystal. Spectroscopic ellipsometric data is simultaneously acquired from the depositing film. Generally, quartz crystal microbalance measures an areal mass density which is related to the density and the geometric thickness of the film, while in-situ spectroscopic ellipsometric can determine either thickness or refractive index for very thin film. Therefore, a reasonable assumption for the density of the film must be made in order to determine the thickness. By using these two techniques in combination, we can find the actual mass condensed at different temperatures. Our findings for the optical constants of materials condensed from different bulk compounds as a function of temperature will be presented.

**EL-ThP5 Combined Electrochemical Impedance Spectroscopy and In Situ Spectroscopic Ellipsometry of Anodically Grown SiO<sub>2</sub>, E.A. Montgomery, University of Nebraska - Lincoln, T.E. Tivald, J.A. Woollam Co., Inc., E. Schubert, M. Schubert, University of Nebraska - Lincoln, C. Beasley, Gamry Instruments, C. Briley, University of Nebraska - Lincoln**

Electrochemical oxidation of silicon (n-type) at room temperature in a mixture of ethylene glycol, water and potassium nitrate has been performed by applying constant current densities to prepare thin SiO<sub>2</sub> layers. In-situ Electrochemical Impedance Spectroscopy (EIS) and Spectroscopic Ellipsometry (SE) are employed during the SiO<sub>2</sub> film growth. Using EIS and SE techniques in-situ one is able to monitor the capacitive changes and also the film thickness change of the oxide. The thickness of the oxides is also measured ex-situ before and after growth using SE. Equivalent circuit models corresponding to the electrolyte-oxide-silicon interfaces and optical

models are fit to EIS and SE data respectively, to gain insight into the quality of the anodically grown SiO<sub>2</sub>.

**EL-ThP7 In Situ Spectroscopic Ellipsometry of Nanoscale Germanium Films Deposited via High Power Impulse Magnetron Sputtering.** *N. Murphy*, Air Force Research Laboratory, *L. Sun*, Air Force Research Laboratory and General Dynamics Information Technology, *A. Waite*, Air Force Research Laboratory and Universal Technology Corporation, *J. Jones*, *R. Jakubiak*, Air Force Research Laboratory

Germanium films were deposited on both glass and silicon substrates using high power impulse magnetron sputtering (HiPIMS). Throughout the deposition process, the optical constants and thicknesses were measured and recorded via *in-situ* spectroscopic ellipsometry. Preliminary analysis of the films' optical behavior has indicated that the refractive index is highly sensitive to changes in thickness. As film thickness increases from 100 to 500 Å, the refractive index displays the tendency to slowly decrease due to void porosity, lack of crystalline order and surface roughness. The preservation of the refractive index seen in the HiPIMS deposited Ge films is a direct result of their high density and low void fraction, following the relationship between density and refractive index as given by the Gladstone-Dale approximation.

# Friday Morning, November 4, 2011

## Spectroscopic Ellipsometry Focus Topic

Room: 209 - Session EL+AS+EM+MS+PS+TF-FrM

## Spectroscopic Ellipsometry: Future Directions and New Techniques

Moderator: H. Wormeester, MESA, The Netherlands

8:20am **EL+AS+EM+MS+PS+TF-FrM1 Current Trends and Future Outlook for Spectroscopic Ellipsometry**, *J.N. Hilfiker, B. Johs, C.M. Herzinger, T.E. Tiwald, J.A. Woollam Co., Inc.* **INVITED**

This talk reviews the significant developments in spectroscopic ellipsometry (SE) in areas including extending spectral range, improving accuracy, and enhancing speed. Current SE applications owe much to hardware and software developments of the past. Thus, today's research efforts may reach full potential for applications years or even decades from now. With this in mind, we point to the current state-of-the-art and what this may mean for future SE applications.

Three important areas will be explored. First, there has been a continual trend to expand SE wavelength range. This has included extensions to both shorter and longer wavelengths. For the latter, there is current development into the THz. More immediate benefit may come from smaller SE extensions from the ultraviolet to the near infrared. For example, further near-infrared extensions help to characterize modern transparent conductive oxides (TCOs), used in both inorganic and organic photovoltaic stacks.

Second, we look at the search for improved SE accuracy. Substantial improvements have come with the development of new ellipsometer technologies, progressing from rotating analyzer/polarizer to rotating compensator and now dual-rotating compensator ellipsometers. In addition to improved accuracy, this technology provides advanced measurements, including the complete Mueller-matrix. This will open SE characterization to new applications of anisotropic, nanostructured, and even patterned thin films. Accuracy enhancements must be compatible with the expanding SE spectral range. Infrared SE has overcome many non-ideal optical components to provide measurements competitive to standard FTIR measurements.

Third, we look at the quest for improved measurement speed. This development is constrained by the previous requirements. The benefits of a wide spectral range generally outweigh speed requirements; otherwise laser-based ellipsometry would still have a strong foothold. Thus, compromises are made depending on application. Current instrumentation typically utilizes detector arrays for multi-channel SE measurements.

To conclude, we will look at the SE outlook and how it may take advantage of wavelength range, accuracy, and speed. In-line and in-situ SE measurements show special promise. Significant improvements in instrumentation, computing speed, and software are now making these applications more feasible. In addition, there are novel ideas to provide sample access and overcome non-ideal measurement conditions for in-line and in-situ SE. Significant progress in many different areas promises to extend ellipsometry into new areas – many of which are being studied by researchers today.

9:00am **EL+AS+EM+MS+PS+TF-FrM3 THz Optical Hall-effect and MIR-VUV Ellipsometry Characterization of 2DEG Properties in a HfO<sub>2</sub> Passivated AlGaIn/GaN HEMT Structure**, *S. Schöche, U. of Nebraska - Lincoln, J. Shi, Cornell U., A. Boosalis, P. Kühne, U. of Nebraska - Lincoln, C.M. Herzinger, J.A. Woollam, J.A. Woollam Co., Inc., W.J. Schaff, L.F. Eastman, Cornell U., V. Darakchieva, Linkoping U., Sweden, M. Schubert, T. Hofmann, U. of Nebraska - Lincoln*

Nitride based high electron mobility transistors (HEMT) utilize the formation of a two-dimensional electron gas (2DEG) at the interface between GaN and AlGaIn due to a difference in spontaneous polarization. It is known that surface traps significantly influence the electrical properties of this 2DEG. Accurate knowledge about the influence of surface passivation on the channel properties is crucial. The device performance is governed by the mobility, the sheet charge density, and the effective mass of electrons in the 2DEG. These parameters are typically determined by electrical Hall effect (EHE), Shubnikov-de Haas (SdH), or cyclotron resonance (CR) measurements. Commonly these experiments require very low temperatures and high magnetic fields. Complex contact configurations are required for SdH and EHE and the ability to locate the 2DEG and possible parallel current paths is limited.

We present non-contact, optical measurements of free-charge carrier mobility, sheet density, and effective mass parameters of the 2DEG for a HfO<sub>2</sub>-passivated AlGaIn/GaN HEMT structure at room temperature.

Spectroscopic ellipsometry in the spectral range from THz and Mid-IR to VUV and THz optical Hall-effect (generalized ellipsometry in magnetic field) (OHE) are employed.

The MIR measurements are performed for analysis of the heterostructure constituents' layer thickness, phonon modes, and volume free charge carriers. The phonon mode parameters were found to be in excellent agreement with literature values and the existence of significant volume charge carrier concentrations could be excluded. NIR to VUV ellipsometry is used to determine the thickness of the thin top layers. From a line-shape analysis in the VUV spectral range the optical constants of the HfO<sub>2</sub> passivation layer could be extracted. An amorphous structure of the HfO<sub>2</sub> passivation layer could be confirmed by comparison with existing studies in literature.

OHE in the THz spectral range is performed for characterization of the 2DEG channel parameters. A classical Drude model for free charge carrier contribution to the dielectric function was applied to determine the sheet density, the carrier mobility, and the effective mass of the 2DEG electrons. The electron effective mass of  $(0.22 \pm 0.04) m_0$  extracted here using OHE corroborates the values found in previous SdH and CR studies. The values for the high-frequency sheet density and carrier mobility obtained by the optical investigations in the THz spectral range are in excellent agreement with results from dc EHE measurements indicative within linear Boltzmann transport theory for frequency-independent carrier scattering mechanisms of the 2D carrier distribution.

9:20am **EL+AS+EM+MS+PS+TF-FrM4 Vector-Magneto-Optical Generalized Ellipsometry on Sculptured Thin Films**, *D. Schmidt, C. Briley, E. Schubert, M. Schubert, University of Nebraska - Lincoln*

Sculptured thin films are self-organized and self-assembled three-dimensional nanostructures with tunable geometries. These artificial nanostructured thin films exhibit highly anisotropic physical properties, which mainly depend on their specific geometry.

Slanted, highly-spatially coherent, columnar nanostructure samples were prepared by glancing angle electron-beam deposition. Glancing angle deposition is a bottom-up fabrication technique that employs a physical vapor deposition process at oblique angles where the trajectory of the incoming particle flux is not parallel to the substrate normal. The technique allows to engineer the columnar film structure and is today amongst the most promising self-organized fabrication processes in micro- and nanotechnology.

We present and discuss the novel approach of vector-magneto-optical generalized ellipsometry on ferromagnetic permalloy nanostructured thin films carried out at room temperature. Investigations have shown that the metal alloy thin films are highly transparent, reveal strong form-induced birefringence, and exhibit intriguing magneto-optical anisotropy. Spatial magnetization orientation hysteresis and magnetization magnitude hysteresis properties are studied using a three-dimensional Helmholtz coil arrangement. This particular octupole setup allows for arbitrary magnetic field directions at the sample position with field strengths up to 200 mT while optical access is granted for reflection and transmission-type ellipsometry measurements. Analysis of data obtained within this unique vector-magneto-optic setup reveals magnetization anisotropy of the permalloy slanted nanocolumns and gives insight into switching behavior of confined magnetic domains.

9:40am **EL+AS+EM+MS+PS+TF-FrM5 THz Dielectric Anisotropy of Metal Slanted Columnar Thin Films**, *T. Hofmann, D. Schmidt, A. Boosalis, P. Kühne, R. Skomski, University of Nebraska-Lincoln, C.M. Herzinger, J.A. Woollam, J.A. Woollam Co., Inc., M. Schubert, E. Schubert, University of Nebraska-Lincoln*

Sculptured thin films (STFs) present an interesting class of self-organized, artificially made materials with three-dimensional, highly spatially coherent arrangements of nanostructures. Contemporary interest in materials for terahertz (THz) electronic, optoelectronic, and optical applications is redrawing attention to STFs that may enable designed optical properties for the THz frequency region.

We report on the anisotropic optical dielectric functions of a metal (cobalt) slanted columnar thin film deposited by electron-beam glancing angle deposition for the THz frequency domain using generalized spectroscopic ellipsometry. A simple anisotropic Bruggeman effective medium dielectric function homogenization approach is successfully employed to describe the observed optical response. This approach describes isolated, electrically conductive columns which render the thin film biaxial (orthorhombic). The anisotropy induced by the columnar film structure is very large. The anisotropic Bruggeman effective medium approach predicts upon slight modifications of Drude, fraction and/or depolarization parameters that



targeted optical properties of STF in the THz range can be achieved by variation of slanting angle, lateral column density, and material.

10:00am **EL+AS+EM+MS+PS+TF-FrM6 A Compact High-speed Spectroscopic Ellipsometer**, G. Chin, ULVAC Inc., Japan

Recently, we developed a compact, high-speed spectroscopic ellipsometer. It analyzes the spectrums obtained from the polarization interference occurring between two multiple-order retarders which snapshot the wavelength distribution of the sample's spectroscopic polarization parameters. This innovative spectroscopic ellipsometer can measure the thickness and optical constants of thin films at a dramatically fast speed. Its acquisition time is as short as 10 ms. It does not require the conventional complex mechanical or active components for polarization-control, such as a rotating compensator and an electro-optical modulator. It can open great opportunities for new applications of the spectroscopic ellipsometry in which the compactness, the simplicity and the rapid response are extremely important. For example, it was integrated into the deposition tool and successfully measured thin films in the vacuum chamber.

This paper describes the principle, system configuration and our innovative efforts on developing the compact high-speed spectroscopic ellipsometer. Some typical application data will be also introduced, such as in line and in situ measurements for photovoltaic, flat panel display and semiconductor industries.

10:20am **EL+AS+EM+MS+PS+TF-FrM7 Ellipsometry Porosimetry (EP): In Situ Spectroscopic Ellipsometry Measurements Coupled with Pressure Controlled Adsorption of Organic Vapors to Study Properties of Nano-Porous Thin Films**, J.P. Piel, L. Kitzinger, A. Bondaz, C. Defranoux, SEMILAB-SOPRALAB, France

Ellipsometric porosimetry (EP) is a non contact, non destructive technique that is cited as a reference technique for porous thin film analysis [1, 2]. As it is based on a spectroscopic ellipsometric measurement, the technique allows the precise determination of the refractive indices and thickness of the porous films. The advantage of these EP tools is that the combination of this well established spectroscopic ellipsometric (SE) technique with a suitably adapted adsorption chamber permits access to all the information obtained by classic adsorption experiments (e.g. BET) on thin films with an excellent sensitivity. Information such as open and closed porosity, pore size distribution etc... can be thus obtained.

In addition the EP allows access to a multitude of information that the classic equipment does not. For example, Spectroscopic Ellipsometry allows to follow the variation of the sample thickness during the adsorption experiment, leading to the determination of the Young's Modulus for the thin films. This will be presented. The technique is highly sensitive to the detection of interfaces; it is thus possible to detect a porosity gradient or to study a multilayer structure and thereafter simultaneously plot the two corresponding adsorption isotherms [3]. In the same manner, the instrument permits the use of a range of different gases adsorptive in order to tailor the probe molecule to the morphology and to the chemistry of the porous layer at ambient temperature [4]. We thus obtain information on the chemistry of the pores within the layer, before, during and after the adsorption experiment. Recent developments include the implementation of the FTIR interferometer SE extension to the EP system. It allows a precise characterization of the chemistry of the pores within the layer. We thus obtain information on the chemical bonds present in the layers before, during and after the adsorption experiment. Preliminary results will be presented.

Specifically, this fundamental technique permits the thorough characterization of porous thin film samples. We will demonstrate some of the different features of the EP technique with regards to the morphological and chemical properties of the porous thin films. Additionally, we will illustrate the technique for various thin film applications such as solgel thin films, nanofilms for catalysis, photovoltaic cells, fuel cells, optical sensors, and bio-compatible materials to name but a few.

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Hwang, S.Y.: EL-ThP2, **6**

## — I —

Ianno, N.: EL+AS+EM+MS+PS+TF-ThA8, **4**  
Ianno, N.J.: EL-ThP4, **6**

## — J —

Jakubiak, R.: EL-ThP7, **7**  
Järrendahl, K.: EL+AS+EM+MS+PS+TF-ThM4, **1**;  
EL-ThP3, **6**  
Johs, B.: EL+AS+EM+MS+PS+TF-FrM1, **8**  
Jones, J.: EL-ThP7, **7**

## — K —

Kariis, H.: EL+AS+EM+MS+PS+TF-ThM4, **1**  
Kasputis, T.: EL+AS+EM+MS+PS+TF-ThM5, **1**  
Kessels, W.M.M.: EL+AS+EM+MS+PS+TF-ThA7, **3**  
Kim, T.J.: EL-ThP2, **6**  
Kim, Y.D.: EL-ThP2, **6**  
Kitzinger, L.: EL+AS+EM+MS+PS+TF-FrM7, **9**;  
EL+AS+EM+MS+PS+TF-ThM10, **2**  
Knoops, H.C.M.: EL+AS+EM+MS+PS+TF-ThA7, **3**  
Kozhukhov, A.S.: EL-ThP1, **6**  
Kruchinin, V.N.: EL-ThP1, **6**  
Kühne, P.: EL+AS+EM+MS+PS+TF-FrM3, **8**;  
EL+AS+EM+MS+PS+TF-FrM5, **8**

## — L —

Lai, R.Y.: EL+AS+EM+MS+PS+TF-ThM6, **2**  
Landin, J.: EL+AS+EM+MS+PS+TF-ThM4, **1**  
Leick, N.: EL+AS+EM+MS+PS+TF-ThA7, **3**  
Levi, D.: EL+AS+EM+MS+PS+TF-ThA1, **3**  
Lucatorto, T.B.: EL+AS+EM+MS+PS+TF-ThM9, **2**

## — M —

Mackus, A.J.M.: EL+AS+EM+MS+PS+TF-ThA7, **3**  
Magnusson, R.: EL-ThP3, **6**  
Marsillac, S.: EL+AS+EM+MS+PS+TF-ThA4, **3**  
Marvel, R.E.: EL+AS+EM+MS+PS+TF-ThA11, **4**  
Montgomery, E.A.: EL-ThP5, **6**  
Motyka, M.A.: EL+AS+EM+MS+PS+TF-ThA10, **4**  
Murphy, N.: EL-ThP7, **7**

## — N —

Nag, J.: EL+AS+EM+MS+PS+TF-ThA11, **4**

## — O —

Ogieglo, W.: EL+AS+EM+MS+PS+TF-ThA9, **4**

## — P —

Pannier, A.K.: EL+AS+EM+MS+PS+TF-ThM5, **1**  
Piel, J.P.: EL+AS+EM+MS+PS+TF-FrM7, **9**;  
EL+AS+EM+MS+PS+TF-ThM10, **2**  
Podraza, N.J.: EL+AS+EM+MS+PS+TF-ThA10, **4**;  
EL+AS+EM+MS+PS+TF-ThA3, **3**  
Pokrovsky, L.D.: EL-ThP1, **6**  
Pu, J.: EL-ThP4, **6**

## — R —

Ramana, C.V.: EL-ThP1, **6**  
Ranjan, V.: EL+AS+EM+MS+PS+TF-ThA4, **3**  
Rauch, S.: EL+AS+EM+MS+PS+TF-ThM3, **1**  
Richter, L.J.: EL+AS+EM+MS+PS+TF-ThM9, **2**  
Rodenhausen, K.B.: EL+AS+EM+MS+PS+TF-ThM5, **1**; EL+AS+EM+MS+PS+TF-ThM6, **2**

## — S —

Sandström, P.: EL-ThP3, **6**  
Schaff, W.J.: EL+AS+EM+MS+PS+TF-FrM3, **8**  
Schmidt, D.: EL+AS+EM+MS+PS+TF-FrM4, **8**;  
EL+AS+EM+MS+PS+TF-FrM5, **8**;  
EL+AS+EM+MS+PS+TF-ThA8, **4**;  
EL+AS+EM+MS+PS+TF-ThM5, **1**  
Schöche, S.: EL+AS+EM+MS+PS+TF-FrM3, **8**  
Schubert, E.: EL+AS+EM+MS+PS+TF-FrM4, **8**;  
EL+AS+EM+MS+PS+TF-FrM5, **8**;  
EL+AS+EM+MS+PS+TF-ThA8, **4**; EL-ThP5, **6**  
Schubert, M.: EL+AS+EM+MS+PS+TF-FrM3, **8**;  
EL+AS+EM+MS+PS+TF-FrM4, **8**;  
EL+AS+EM+MS+PS+TF-FrM5, **8**;  
EL+AS+EM+MS+PS+TF-ThA8, **4**;  
EL+AS+EM+MS+PS+TF-ThM5, **1**;  
EL+AS+EM+MS+PS+TF-ThM6, **2**; EL-ThP5, **6**  
Shi, J.: EL+AS+EM+MS+PS+TF-FrM3, **8**  
Skomski, R.: EL+AS+EM+MS+PS+TF-FrM5, **8**  
Sun, L.: EL-ThP7, **7**

## — T —

Tarrio, C.: EL+AS+EM+MS+PS+TF-ThM9, **2**  
Tiwald, T.E.: EL+AS+EM+MS+PS+TF-FrM1, **8**;  
EL-ThP5, **6**

## — U —

Uppireddi, K.: EL+AS+EM+MS+PS+TF-ThA6, **3**

## — V —

Vemuri, R.S.: EL-ThP1, **6**

## — W —

Waite, A.: EL-ThP7, **7**  
Wang, H.: EL+AS+EM+MS+PS+TF-ThM5, **1**;  
EL+AS+EM+MS+PS+TF-ThM6, **2**  
Weber, J.W.: EL+AS+EM+MS+PS+TF-ThA7, **3**  
Weber, M.J.: EL+AS+EM+MS+PS+TF-ThA7, **3**  
Woollam, J.A.: EL+AS+EM+MS+PS+TF-FrM3, **8**;  
EL+AS+EM+MS+PS+TF-FrM5, **8**  
Wormeester, H.: EL+AS+EM+MS+PS+TF-ThA9, **4**

## — Y —

Yan, L.: EL+AS+EM+MS+PS+TF-ThA6, **3**  
Yoon, J.J.: EL-ThP2, **6**

## — Z —

Zhou, F.: EL-ThP4, **6**