Applied Surface Science
Room: C2 - Session AS+EM+MS+TF-MoM

Spectroscopic Ellipsometry I
Moderator: M.S. Wagner, Proctor and Gamble

8:20am **AS+EM+MS+TF-MoM1 Optical Properties of Bulk GaSe and InSe Single Crystals**, ***S.G. Choi***, National Renewable Energy Laboratory, *C. Martinez-Tomas, V. Munoz Sanjose,* Universitat de Valencia, Spain, *D.H. Levi*, National Renewable Energy Laboratory

III-VI compounds generally crystallize in layered-structures characterized by strong covalent interactions *within* the layers but weak Van der Waals binding *between* the layers. This unique structural characteristic has made III-VI compounds attractive for their potential applications in nonlinear optics. Among these compounds, in particular, InSe has been considered as a promising candidate for thin film photovoltaic (PV) material owing to its energy bandgap, optical and transport properties. Recently, high-quality epitaxial InSe thin films have been grown on GaSe substrates, and PV device structures containing *n*-InSe and *p*-GaSe have been successfully fabricated [1].

In order to design and optimize a high-performance PV device structure, knowledge of optical properties of constituent materials over a wide spectral range is required. However, large discrepancies were found in the properties of GaSe and InSe available in the literature, which have been measured mostly by reflectance methods with the Kramers-Kronig transformation employed to obtain the dielectric functions. Here, we present ellipsometrically determined pseudodielectric function $\langle\varepsilon\rangle=\langle\varepsilon_1\rangle+i\langle\varepsilon_2\rangle$ spectra from 0.73 to 6.45 eV of bulk GaSe (ε-phase) and InSe (γ-phase) single-crystals grown by a vertical Bridgman method. The surfaces with minimum overlayers were obtained by peeling off the top few layers from the sample surface and ellipsometric measurements were immediately followed under flowing N_2 environment, which yields good approximations to the intrinsic dielectric responses. The measured spectra exhibited a number of interband-transition critical-point structures, and their energy values were obtained precisely from numerically calculated second-energy-derivatives of $\langle\varepsilon\rangle$ assuming the parabolic-band critical-point model.

Data obtained in this work can be used to model PV device structures utilizing GaSe and InSe, and the critical-point energies determined will be useful for theoreticians to perform fine band structure calculations of III-VI compounds.

The work done at Universitat de València was supported in part by the Spanish Project MAT2007-06841. This abstract is subject to U.S. government rights.

[1] J.F. Sánchez-Royo, J. Appl. Phys. 90, 2818 (2001).

8:40am **AS+EM+MS+TF-MoM2 Ellipsometric Porosimetry for the Microstructure Characterization of Plasma-Deposited SiO_2-Like Films**, ***M. Creatore***, *N.M. Terlinden, G. Aresta, M.C.M. van de Sanden*, Eindhoven University of Technology, The Netherlands

SiO_2 layers have been deposited from Ar/O_2/hexamethyldisiloxane mixtures in a remote expanding thermal plasma setup enabling a good control of both the ion flux (by changing the deposition chemistry and the arc plasma parameters) as well as the ion energy. This latter is achieved by an additional rf substrate biasing or a tailored ion biasing technique, i.e. a low frequency pulse-shaped bias. The role of the ion energy and ion-to-growth flux ratio on the film microstructure and densification at low substrate temperature (100ºC) has been investigated by means of ellipsometric porosimetry. This technique monitors the refractive index change due to the adsorption (and desorption) of ethanol vapors in the volume of macro-meso-micro pores in the SiO_2 layer. From the analysis of the adsorption isotherm and the presence of hysteresis during the desorption step as a function of the equilibrium partial pressure, the open porosity in the layer can be determined. It is found that both biasing techniques lead to densification of the deposited layer, which experiences a transition from micro-/ mesoporosity to microporosity and eventually non-porosity, as function of the increasing ion energy. Although both biasing techniques lead to a comparable critical ion energy value per deposited SiO_2 unit (about 100 eV), the ion-to-growth flux ratio and ion energy are not found to be interchangeable parameters. In fact, in the case of the rf bias, the meso- and large micropores are first affected leading to a quantitative decrease of porosity, i.e. from 11% to 3% at an ion energy less than 20 eV. A further increase in ion energy eventually reduces the presence of smaller micropores leading to non porous films at energy of 45 eV. When the pulse-shaped biasing technique is adopted, the micro- and mesopores are simultaneously affected over the whole range of available ion energy, leading to a non porous layer only at very high energy values, i.e. 240 eV. This difference is attributed to the increasing ion-to-growth flux ratio accompanying the rf biasing, as a consequence of the rf plasma generation in front of the substrate.

9:00am **AS+EM+MS+TF-MoM3 Industrial Applications of Spectroscopic Ellipsometry**, ***J.A. Woollam***, J.A. Woollam Company, Inc., *J.N. Hilfiker, P. He*, J.A. Woollam Company Inc. **INVITED**

Spectroscopic Ellipsometry (SE) has been used for decades for basic research on surfaces and thin films. Hundreds of articles, review papers, and books describe SE use in physics, chemistry and surface and materials engineering. Far less is available describing industrial applications because companies gain competitive advantage using SE and are not motivated to publish.

Without revealing anyone's proprietary information, this talk reviews examples of SE use in industry. This involves both production quality control (QC), and product development. Best known is SE for QC in integrated circuit manufacturing. Others include integrated circuit critical dimension (CD) metrology, read-write heads, display technologies, optoelectronics, photovoltaics (crystalline and thin film), optical coatings, web-coaters, wear surfaces, and protective coatings. Industrial SE applications include ex-situ, in-situ, and in-line metrology.

9:40am **AS+EM+MS+TF-MoM5 Spectroscopic Ellipsometry Studies of Sputtered Vanadium Oxide Thin Films**, ***N.J. Podraza***, *B.D. Gauntt, M.A. Motyka, E.C. Dickey, M.W. Horn*, The Pennsylvania State University

Vanadium oxide (VO_x) thin films have been used for the last twenty years as the imaging material in uncooled infrared imaging devices. The important material properties for this application are a high thermal coefficient of resistance (TCR), controllable resistivity (ρ), low electrical noise and process compatibility with standard IC fabrication. However, vanadium can adopt many different oxidation states, yielding a number of stable metal oxides, which can lead to difficulties in reliable and consistent device fabrication. In this work, VO_x thin films were fabricated via pulsed-DC magnetron sputtering in an argon and oxygen atmosphere under variable total pressure and oxygen-to-argon ratio deposition conditions in order to investigate the variability in desired material properties. In situ real time spectroscopic ellipsometry (RTSE) has been applied to stuffy films prepared under variable deposition conditions in order to evaluate the microstructural evolution of VO_x during film growth and changes occurring to the surface and bulk material upon initial exposure to atmosphere. These films were characterized ex situ using a number of complementary techniques including, Rutherford backscattering spectroscopy (RBS) in order to obtain the oxygen content, x; transmission electron microscopy (TEM) to determine film crystallinity; glancing incidence X-ray diffraction (GIXRD) was used to ensure localized measurements from the TEM were representative of the entire film; and I-V curve measurements as a function of temperature were used to determine the film resistivity and TCR. By varying deposition conditions, the film resistivity was varied over seven orders of magnitude from ~10^{-3} to 10^4 Ω-cm and the TCR spanned from -0.1 to -3.5 %/K. The growth evolution, complex dielectric function spectra ($\varepsilon = \varepsilon_1 + i\varepsilon_2$), and structure are correlated to these electrical properties. Films produced at low oxygen-to-argon ratios exhibit nanocrystalline V, V_2O, and VO phase material dependent on the specific deposition conditions, while films produced at higher oxygen-to argon ratios are amorphous. In both the nanocrystalline and amorphous phases, features in e obtained from spectroscopic ellipsometry have been shown to correlate with the oxygen content and resistivity and RTSE studies have been used to monitor changes occurring at the film / ambient interface after the vanadium oxide is exposed to air. This array of techniques were used to establish the roles deposition parameters play in the final structure and composition of each film, as well as to determine the resulting effects of these characteristics on the electronic transport and optical properties.

10:00am **AS+EM+MS+TF-MoM6 Real Time Spectroscopic Ellipsometry Studies of Si:H and Ge:H Thin Films for Microbolometer Applications**, ***D. Saint John***, *E.C. Dickey, N.J. Podraza*, The Pennsylvania State University

Thin film hydrogenated silicon (Si:H) and germanium (Ge:H) have been of wide interest as thin film semiconducting materials, and are now of growing interest for use in infrared sensing uncooled microbolometers, although the impact of the growth evolution and structure on device performance is only beginning to be determined. Ideal properties for incorporation of these layers in microbolometers include: a high temperature coefficient of resistance (TCR); controllable resistivity (ρ); low 1/f noise within frequencies of interest; and process compatibility with standard IC

fabrication. In this work, n- and p-type doped Si:H and undoped Ge:H thin films have been prepared by plasma enhanced chemical vapor deposition (PECVD) with resulting resistivities ranging from 1.5 to 2500 Ω cm and TCR ranging from –0.8 to –4.0 %/K and studied using real time spectroscopic ellipsometry (RTSE). These films, monitored in situ during growth by RTSE, have been shown to exhibit changes in microstructure as a function of deposition conditions. For example, films prepared at low hydrogen dilution may remain amorphous throughout growth (a-Si:H), while films prepared at higher dilution may initially grow as amorphous until a bulk layer thickness where microcrystallites nucleate and eventually coalesce into a single-phase microcrystalline layer (μc-Si:H). A combination of in-situ RTSE, transmission electron microscopy (TEM), and electrical measurements (ρ, TCR, 1/f noise) have been used to study the effects of deposition conditions on the resulting microstructure during film growth and the dependence of the electrical properties on this microstructure. Studies of p-type a-Si:H have shown that both TCR and ρ increase with hydrogen dilution for fixed doping gas-to-silane ratio, which suggests that optimizing the TCR for a film of a given resistivity may potentially be obtained by varying both the hydrogen and doping gas dilutions. n-type a-Si:H and mc-Si:H films were evaluated in order to quantify changes in TCR and r resulting from microstructural differences (a-Si:H: ρ= 250 Ω cm, TCR = –3.8 %/K; μc-Si:H: ρ = 1.5 Ω cm, TCR = –0.8 %/K). Growth evolution studies of undoped Ge:H films prepared under variable hydrogen dilution conditions show transitions from amorphous to microcrystalline material at higher hydrogen dilution and relatively high TCR values ranging from –2.2 to –3.6 %/K as dilution is increased within the amorphous growth regime.

10:40am **AS+EM+MS+TF-MoM8 Non-destructive Determination of Spatial Distributions of Free-Charge-Carriers in Low Doped Semiconductors using THz Ellipsometry**, ***T. Hofmann***, University of Nebraska-Lincoln, *C.M. Herzinger,* J. A. Woollam Co. Inc., *M. Schubert*, University of Nebraska - Lincoln

The non-contact and non-destructive optical determination of spatial distributions of free-charge-carriers in low doped semiconductor homo- and heterojunctions addresses fundamental physical properties of device related structures. However, the optical characterization of low density free-charge-carriers, particularly for hole densities with their intrinsically lower mobility parameters than electron densities is very challenging. For low carrier densities the plasma frequencies are located at within the terahertz (THz) spectral region and measurements of plasma frequency properties in a at THz frequencies have been used for the determination of free-charge-carrier properties in single crystals (e.g. [1,2]). The characterization of free-charge-carrier properties in low-doped homo- and heterostructures remains a challenge.

Here we report on the non-invasive optical measurement of hole diffusion profile in a p-p^+ Silicon homojunction by spectroscopic ellipsometry in the terahertz (0.2 to 1.5 THz) and mid-infrared (9 to 50 THz) spectral regions. In the terahertz region a surface guided wave resonance with transverse-electrical polarization is observed at the boundary of the p-p^+ homojunction, and which is found to be extremely sensitive to the low-doped *p*-type carrier concentration as well as to the hole diffusion profile within the p-p^+ homojunction. Effective mass approximations allow determination of homojunction hole concentrations as p=2.9x10^{15} cm^{-3}, p^+=5.6x10^{18} cm^{-3}, and diffusion time constant D_t = 7.7x10^{-3} μm^2, in agreement with previous electrical investigations.

[1] D. Grischkowsky, Soren Keiding, Martin van Exter, and Ch. Fattinger, J. Opt. Soc. Am. B **7**, 2006 (1990).

[2] M. Herrmann, M. Tani, K. Sakai, and R. Fukasawa, J. Appl. Phys. **91**, 1247 (2002).

11:00am **AS+EM+MS+TF-MoM9 Ellipsometric Depth Profiling of Polymer-Blend Films for Organic Electronics and Photovoltaics**, ***L.J. Richter***, *D.S. Germack, D.M. DeLongchamp, D.A. Fischer, V.M. Prabhu, D.J. Gundlach,* National Institute of Standards and Technology, *J.E. Anthony,* University of Kentucky, *N. Shin, D. Yoon*, Seoul National University, Korea

Interest has emerged in the development of devices based on organic materials and low energy, low cost, roll-to-roll fabrication techniques. Two specific target applications have received particular attention: organic transistors to enable macroelectronics (large area displays, RFID tags, etc.) and organic photovoltaics. Common to both applications is the development of optimal inks: for macroelectronics, small crystallizable molecules in an inert polymer binder, for photovoltaics, fullerene based acceptors blended in an active polymer donor. Critical to a proper understanding of the device performance is an understanding of the vertical profile of the fabricated film. For thin film transistors, segregation of the small molecule semiconductor to the interfaces is essential. For PV, segregation of the fullerene can be both advantageous (if at the cathode) and deleterious (if at the anode). Spectroscopic ellipsometry studies of the vertical profile are daunting, as the systems are in general anisotropic with individual component spectra that are strongly dependent on the local order. We present SE studies of both TFTs and OPV devices using a multiple sample approach to remove correlations in the resultant models. The optical depth profiles are compared to both NEXAFS studies of the interface composition and neutron reflectivity studies of similar processed films. We find good agreement between the SE and less model dependent techniques. The studies illuminate general principles of the influence of interfacial free energy on the resultant segregation of the species.

11:20am **AS+EM+MS+TF-MoM10 Thickness Variations Determined by Spectroscopic Ellipsometry in Organometallic Chemical Vapor Deposition: Connection to Growth Processes**, ***X. Liu***, *D.E. Aspnes*, North Carolina State University

Lateral thickness variations are commonly observed for films deposited by organometallic chemical vapor deposition (OMCVD). The variations are typically systematic near boundaries between growth and adjacent surfaces, for example masks. We use spectroscopic ellipsometry to study thickness variations for the heteroepitaxy of GaP by OMCVD on (001) GaAs, thermally generated SiO_2, (001) Si, and nanoscopically roughened Si surfaces using trimethylgallium (TMG) and phosphine (PH_3) sources, showing that the variations provide information about details of OMCVD growth that would be difficult to obtain in any other way. Our reference surface is the polycrystalline GaP inadvertently deposited on the Mo susceptor that surrounds the 2 in. wafers. We find that the thicknesses of the deposited GaP films increase or decrease accurately exponentially toward the edge. Using an analytic Green-function approach based on the one-dimensional diffusion equation, we show that for our growth conditions (4 Torr chamber pressure), the exponential thickness variations are due to differences in chemical reactivities of the various surfaces, especially on the different catalytic effects that they exert on PH_3 decomposition. The results show directly that different parts of the surface, including the susceptor, are in constant contact with each other through lateral gas-phase diffusion. The data are explained by assuming that growth occurs via a precursor that is formed by heterogeneous catalysis, largely desorbs, and involves both Ga and P, for example the H-P=Ga-CH_3 dimer adduct. To distinguish this process from selective area growth (SAG), which takes place with a similar configuration, we also solve the SAG problem analytically, in this case using a conformal map. For SAG the thickness near mask edges is found to vary as $r^{-1/2}$, where r is the lateral distance away from the edge. The distinctive differences in these thickness dependences indicate that SAG growth occurs via a different mechanism.

11:40am **AS+EM+MS+TF-MoM11 Optical Anisotropy Induced by Oblique Incidence Ion Bombardment of Ag(001)**, ***H. Wormeester***, *F. Everts, B. Poelsema*, University of Twente, The Netherlands

Oblique incidence ion sputtering has become a widely used method for the creation of highly regular patterns of lines and dots. On a Ag(001) surface oblique incidence sputtering creates a ripple pattern that exhibits plasmonic features. The photon energy of this plasmonic feature depends on the ripple periodicity. The development of these anisotropic features was measured in-situ with the optical technique Reflection Anisotropy Spectroscopy (RAS).

The ion induced nanopatterning was done with 2 keV Ar ions with a flux of a few mA/cm2 in a temperature range of 300 - 420K. With RAS, a periodicity of ripples above 200 nm is measured by a shift in photon energy of the plasmon resonance. Features with a smaller periodicity show a plasmon resonance around 3.65 eV. For very grazing incidence sputtering, 80o polar angle of incidence, only a resonance feature around 3.65 eV is observed. High resolution LEED measurements after sputtering confirm the formation of 1D nanoripples.

For a polar angle of incidence of the ion beam of 70o a shift in the maximum of the plasmon resonance feature is observed. These spectra can be well described within the Rayleigh-Rice description for scattering from a slightly rough surface. The formation of nanoripples, i.e. a roughening of the surface perpendicular to the direction of the ion beam in one direction suffices to describe the measured optical data. The evolution of the rms, wavelength and wavelength distribution of the ion induced nanoripples is obtained from the in-situ measurements. For a polar angle of the ion beam of 61o we find that also the roughening in the direction along the nanorippels has to be taken into account to describe the optical spectra.

Applied Surface Science
Room: C2 - Session AS+EM+MS+TF-MoA

Spectroscopic Ellipsometry II
Moderator: M. Creatore, Eindhoven University of Technology, The Netherlands

2:00pm **AS+EM+MS+TF-MoA1 Spectroscopic Ellipsometry on Protein Layers: Characterization and Sensor Applications**, ***H. Arwin***, Linköping University, Sweden **INVITED**

Ellipsometry is very attractive for studies of biolayers including protein layers. First, its thickness resolution is well below 1 nm which is perfect for protein layers as they typically are composed of nm-sized molecules. Second, ellipsometry can be used in any transparent medium, so it can be applied to solid-liquid interfaces where many bioreactions take place. Third, molecules do not have to be labeled, as required for techniques based on fluorescence or radioactivity. One drawback is that it is not analytic if operated in the visible spectral range and in simple applications one primarily measures the optical mass on a surface. However, with high precision spectroscopic ellipsometry, structural information in protein monolayers can be obtained in some cases and chemical analysis can be performed with infrared ellipsometry.

In this report, the use of various types of ellipsometry for studies of protein layers at air/solid and liquid /solid interfaces are reviewed. Among the methods included are spectroscopic, dynamic, internal reflection and imaging ellipsometry. Two examples of methodology for analysis are discussed in some detail. First we observe that *in situ* studies allow monitoring of the dynamics of protein layer growth. Modeling of layer structure by separation of refractive index and thickness from such *in situ* data recorded during adsorption of fibrinogen layers is presented as well as strategies for evaluation of surface mass density.

In a second example, a model dielectric function (MDF) concept for protein layers in the UV-VIS-IR spectral range is presented. The MDF contains model parameters like resonance energies and broadenings of vibrational structures, e.g. in the amide bands. Changes in these parameters can be monitored and used to assess the conformational state in the protein layer. As an example, studies of thermally induced degradation of fibrinogen layers are presented.

Finally potential sensor applications based on imaging and dynamic ellipsometry utilizing sub-nm thickness resolution are reviewed. The use of surface-plasmon resonance enhancement to increase resolution of internal reflection ellipsometry to pm in thickness will be discussed.

2:40pm **AS+EM+MS+TF-MoA3 Anisotropic Optical and Magneto-Optical Properties of Sculptured Thin Films**, ***D. Schmidt***, *T. Hofmann, A. Kjerstad, E. Schubert, M. Schubert*, University of Nebraska-Lincoln

Three-dimensional structure design on the nanoscale is in the focus of modern material science and engineering because intriguing applications are foreseen for such nanostructured films in various fields ranging from optics, electromechanics or electromagnetics. We utilize glancing angle electron-beam deposition, where physical shadowing and varying particle incidence azimuth are exploited for fabrication of three-dimensional individual nanostructures arranged in sculptured thin films with different morphologies. We will present the anisotropic (structure-related) optical and magneto-optical properties of sculptured thin films from cobalt. Generalized spectroscopic ellipsometry is employed to determine the anisotropic principal optical constants of slanted columnar and chiral nanocoil thin films in the spectral range from 400 to 1650 nm. These thin films show strong form birefringence and large dichroism and are distinctly different from their bulk material. We will also discuss why slanted columnar thin films have monoclinic optical properties [1]. Magneto-optic Kerr effect measurements in the polar configuration lead to the determination of the magneto-optical Q-values (Voigt parameters) of such highly anisotropic ferromagnetic sculptured thin films.

[1] D. Schmidt, A. C. Kjerstad, T. Hofmann, R. Skomski, E. Schubert, and M. Schubert, J. Appl. Phys. **105**, XXX (2009).

3:00pm **AS+EM+MS+TF-MoA4 Development of Hybrid Quartz Crystal Microbalance / Ellipsometric Porosimetry for the Characterization of Anisotropic Optical Materials**, ***R.A. May***, *D.W. Flaherty, C.B. Mullins, K.J. Stevenson*, University of Texas at Austin

Ellipsometric porosimetry (EP) relies on monitoring the change in optical properties during vapor adsorption/desorption and has been successfully applied to an array of materials using a variety of adsorbates both in vacuum and at ambient pressures. However, these studies typically avoid the analysis of optically complex systems with strong absorbance or optical anisotropy. Towards this end, a hybrid quartz crystal microbalance/ ellipsometric porosimetry (QCM/EP) technique is being developed to facilitate the study of more complex optical materials and to quantitatively estimate parameters such as porosity, pore size distribution, and surface area. To highlight the power of this hybrid approach thin films of TiO_2 and TiC, have been deposited using reactive ballistic deposition (RBD). RBD provides control over film parameters such as surface area, porosity, pore size, and birefringence through variation of the deposition angle. Combined with the QCM/EP technique these films provide a platform for understanding both novel material properties and the requirements for extracting valid optical constants from anisotropic optical materials.

3:40pm **AS+EM+MS+TF-MoA6 Multichannel Ellipsometry for Thin Film Photovoltaics Applications: From Materials to Solar Cells**, ***R.W. Collins***, *J. Li, M.N. Sestak, J.A. Stoke, L.R. Dahal*, University of Toledo **INVITED**

Second generation or thin film photovoltaics (PV) technologies have achieved the lowest manufacturing costs in the PV industry. These technologies benefit from multichannel ellipsometric analysis for characterization of multilayered thin film materials and deposition processes, specifically for determination of component layer thicknesses and dielectric functions. From such results, predictions of the maximum achievable quantum efficiency of multilayered PV device structures are possible. In this presentation, the current applications and future prospects of multichannel spectroscopic ellipsometry (SE) will be discussed for optical characterization of PV materials and devices during fabrication in the research laboratory as well as for on-line and off-line evaluation in PV module production. First, this review will address the advances in instrumentation for multichannel SE. Second, this review will provide examples of the analysis of real time SE data acquired during film growth to obtain structural parameters and dielectric functions, as well as the subsequent analysis of the resulting dielectric functions in terms of parameterized models to deduce useful information on thin film materials properties. Recent applications to be discussed in this presentation involve (i) the analysis of grain size, strain, and void profiles from the dielectric functions of polycrystalline CdS and CdTe thin films used as heterojunctions in efficient solar cells; (ii) the analysis of amorphous and nanocrystalline volume fraction profiles from the dielectric functions of mixed-phase hydrogenated Si (Si:H) thin films also used in efficient solar cells; and (iii) the determination of interface dielectric functions and losses associated with Ag/ZnO structures used as back-reflectors in efficient thin film Si:H PV devices. In the latter studies, the optical features of confined plasmon resonances can be identified. Methods for dealing with microscopic (sub-wavelength order) and macroscopic (wavelength order) surface and interface roughness will be treated, as will its impact on prospects for analyzing PV device structures on-line during module manufacturing. The ability to extract polarization, depolarization, and irradiance information from the reflected beam by multichannel SE is advantageous in many such PV applications.

4:20pm **AS+EM+MS+TF-MoA8 Universal Behavior of Light Scattering from Self-Affine Fractal Surfaces: A Quantitative Relationship between Roughness and EMA Models**, ***A. Yanguas-Gil***, *B.A. Sperling,* University of Illinois at Urbana-Champaign, *J.R. Abelson*, University of Illinois, Urbana-Champaign

The effective medium approximation (EMA) is typically used to model the influence of roughness on the optical response of a surface or buried interface as measured by ellipsometry. Although the standard assumption of 50% material - 50% void provides useful results, the relationship between the EMA layer thickness and the surface topography is not fully understood. For example, in thin film deposition many authors have found a good correlation between the thickness of the EMA layer and the rms surface roughness measured by AFM, while others have found significant discrepancies between the time evolution of these two parameters.

Using first principles scattering models, we have analyzed the ellipsometric response of surfaces that exhibit a self-affine dependence of surface topography on the lateral scale of measurement. This type of surface

roughness is found for a wide variety of real surfaces, including many deposited thin films. The calculations show that when the surface correlation length evaluated from the height-height correlation function or the power spectral density is much smaller than the incident wavelength, a universal behavior is found in the ellipsometric response. Both the amplitude of the reflected fields in the p- and s-polarizations, and the thickness of the EMA layer, depend on the product of the *rms* surface roughness times the average surface slope. Therefore, the linearity between roughness and the thickness of the EMA layer holds only as long as the average surface slope remains constant. That is the case when the growth obeys the predictions of dynamic scaling theory, i.e., the *rms* roughness and the correlation length change with time as $\sigma \sim t^{\beta}$ and $\xi \sim t^{\beta/\alpha}$, where α and β are the roughness and the growth exponents, respectively. Results are presented for different materials whose optical properties cover a broad range from metals to dielectrics. An important consequence of this universality is that the ellipsometric response is mathematically separable into two independent functions, one depending only on the optical properties of the film and the other only on the surface topography.

4:40pm **AS+EM+MS+TF-MoA9 Numerical Ellipsometry: Thin Absorbing Films Deposited on Opaque Substrates**, ***F.K. Urban***, *D. Barton,* Florida International University*, T.E. Tiwald*, J A Woollam Co.

A major challenge for those utilizing ellipsometry is numerical processing of the measured data. The transcendental, multivalued equations arising from the physics of simple reflection are problematic for the least-squares numerical methods in common use. These early numerical methods require fairly accurate initial estimates, bounding to avoid local minima, and only find solutions at the bottom of a relatively flat numerical topography. Previously we have applied Complex Analysis in the n-k plane to improve visualization of the mathematics and this has led to a growing array of new numerical methods avoiding these difficulties. The work presented here extends these new numerical methods for use beyond transparent substrates to include absorbing substrates. Results show that reflection ellipsometry alone can be sufficient for determination of thin absorbing film thickness and optical properties without the need for additional kinds of measurements.

Numerical processing considering surface layers such as air-formed oxides will also be presented.

5:00pm **AS+EM+MS+TF-MoA10 *In situ* Spectroscopic Ellipsometry As a Versatile Tool to Study Atomic Layer Deposition**, ***E. Langereis***, *H.C.M. Knoops, W. Keuning, A.J.M. Mackus, N. Leick, M.C.M. van de Sanden, W.M.M. Kessels*, Eindhoven University of Technology, The Netherlands

Atomic layer deposition (ALD) is considered as one of the primary candidates for the deposition of ultrathin and conformal films with precise growth control. In this contribution, the merits of using *in situ* spectroscopic ellipsometry (SE) to address various aspects of ALD will be discussed. In particular, the versatility of this all-optical diagnostic will be demonstrated by results obtained on metal oxide (Al2O3, HfO2, Er2O3, TiO2, Ta2O5, and SrTiO3), metal nitride (TiN and TaN*x*), and metal (Pt and Ru) films with thicknesses ranging from 0.1 to 100 nm [1]. By acquiring SE data within a combined photon energy range of 0.75-6.5 eV in between the ALD (half-)cycles and by analyzing the film thickness and the energy dispersion of the optical constants of the films, the layer-by-layer growth and material properties of the films can be studied in detail. It will be shown that the growth rate per cycle and the ALD saturation curves can be determined directly by monitoring the film thickness as a function of the number of cycles, while also the nucleation behavior of the films on various substrates can be probed. Furthermore, it is demonstrated that the energy dispersion relation can provide information on the optical properties, the crystalline phase, and the material composition of the films. For metallic films, electrical properties can be calculated from the Drude absorption yielding insight into the electrical resistivity and electron scattering effects in ultrathin films.

[1] E. Langereis et al., J. Phys. D: Appl. Phys. 42, 073001 (2009).

5:20pm **AS+EM+MS+TF-MoA11 Mueller-Matrix Ellipsometry Studies of Optically Active Structures in Scarab Beetles**, ***K. Järrendahl***, *J. Landin, H. Arwin*, Linköping University, Sweden

Ellipsometry is a valuable tool for general materials characterization but also for optical investigations of complex structures including multilayers, photonic crystals, metamaterials and other artificial materials. The complexity of these kind of structures has during the years promoted the use of spectroscopic, variable angle, generalized and Muller-matrix ellipsometry. In parallel more complex optical models and analysis algorithms have come into use.

Naturally occurring structures may show even higher complexity than artifical structures but with a more narrow range of constituent materials, mainly chitin and various proteins. Many interesting structures are found in insects, especially in butterflies and beetles. Fascinating reflection properties result from intricate photonic structures in their wing scales and cuticles. Currently there is a large interest to explore such functional supramolecular architectures for exploitation in nanotechnology.

Even though the optical properties of natural structures are frequently investigated, ellipsometry rarely has been used to reveal structural and optical properties. In this study, Mueller-matrix spectroscopic ellipsometry is applied in the spectral range of 300 to 1700 nm to investigate structures in the cuticle of Scarab beetles, primarily *Cetonia aurata* (the rose chafer). The cuticle of *Cetonia aurata* is green with a metallic look and reflects circular polarized light. It has been suggested that the circular polarization of this metallic gloss is caused by a helical structure in the chitinous cuticle. We find that the circular polarization effect is limited to the narrow spectral range 470-550 nm and for shorter or longer wavelengths the reflection properties are similar to those from a near-dielectric material. Furthermore, the light reflected from *Cetonia aurata* is left-handed circularly polarized and the beetle thus appears black if viewed through a right-handed circular polarizer. In addition to Mueller-matrix spectroscopic ellipsometry, reflectance and scattering measurements are used to characterize the cuticle of *Cetonia aurata*. Model calculations and parameterization of the nanostructure employing a heliocoidal structure are discussed.

Plasma Science and Technology
Room: A1 - Session PS+MS-MoA

Plasma Challenges at the 22nm Node and Beyond
Moderator: C. Labelle, GLOBALFOUNDRIES

2:00pm **PS+MS-MoA1 Plasma Etch Challenges for 22nm Advanced Logic Development**, ***R. Wise***, IBM **INVITED**

At the 22nm technology node for logic devices many novel semiconductor technologies are being considered, each of which impacts etch process development and control. These technology performance challenges drive increases in carrier mobility (necessitating application of high strain liner and epi materials and reduction in silicon loss budget and gate height scaling), increased packing density (limiting resist trim budgets, increasing CD shrink requirements, and increasing integration of eDRAM), and achieving target resistance and capacitance (necessitating the introduction of porous low-k dielectrics and better profile control). The challenges introduced by these elements on dry etch processes, tooling, and controls is discussed in detail.

Widespread aggressive device scaling beyond lithographic limits require dry etch processes to provide controllable CD reduction to meet design groundrules. In particular, limited improvement in imaging at the 22nm node results in challenges in scaling on the plasma equipment. The implementation of multiple exposure techniques to achieve design rules for several key levels drives additional process control across multiple exposure and etch steps. Reduction in the available mask thickness required to preserve the lithography process window have driven the need for highly selective etch processes, generally at the expense of uniformity, defectivity, and profile of the transferred pattern. Later generation lithographic materials are expected to continue to exhibit increased sensitivity to line edge roughness, and drive additional implementation of novel masking materials. Process and tooling technology needs required to address these imaging challenges are discussed.

2:40pm **PS+MS-MoA3 22nm Technology Manufacturing Challenges - Window for Process Control becomes Smaller and Smaller, Equipment and Material Interaction Becomes Unpredictable and Manufacturing Costs Increase**, ***P. Adam***, GLOBALFOUNDRIES Dresden, Germany **INVITED**

Increasing complexity and smaller and smaller CD for 22 nm technologies will have also a major impact for all plasma supported processes. The limited understanding of plasma and device interaction in existing technologies will further challenge the equipment suppliers to develop solutions for high volume manufacturing fabs. Some examples will be shown to illustrate this statement. Fab Engineers will see unexpected behaviour of materials in process chambers and surprising results of their plasma process on the device itself. A big amount of this will not be seen in the application labs of the equipment suppliers. Part of the problem is availability of appropriate test wafer material which can reflect the final manufacturing situation sufficient enough. Designs from different companies will behave most likely also differently. Therefore equipment suppliers have to move their development process close into the

manufacturing site of the fabs. On the other side, semiconductor fab would like to get a tool and a process ready to go. They don't have the time and the manpower to support this kind of development work for the equipment supplier. All this will drive additional cost for both supplier and costumer.

How we can overcome this situation? Some ideas will be presented highlighting the complexity of the situation and the need for close interaction of all involved parties.

3:40pm **PS+MS-MoA6 Logic Etch Challenges at the 22nm Node and Beyond**, ***V. Vahedi***, *G. Kamarthy, J. Guha, H. Singh*, Lam Research Corporation **INVITED**

Due to increased device integration complexity, there are significant challenges to technology scaling for Logic devices at 22nm and beyond. The issues range from difficulties in scaling device threshold voltage (V_t), and electron and ion mobility enhancements to achieving the proper leakage current for low power devices. Proposed solutions to overcome these challenges include adoption of Metal Gate High-k for threshold voltage and leakage current engineering, to various Strained Silicon techniques to enhance electron and ion mobility, and FinFETs for beyond 22nm technology node. In this presentation, we will review some of challenges associated with front-end logic integration schemes, such as control of Si Recess and Si damage. Si loss and damage after gate etch, spacer etch, and strained Si etch applications can impact source-drain junction depth, and increase device leakage. We will discuss various mechanisms for Si loss and damage, work done by previous authors, what is required at 22nm and beyond, implication for etch and post etch clean, and areas where better understanding is required.

Biomaterial Interfaces
Room: K - Session BI+AS+BM+MS-WeM

Array-Based Sensors and Diagnostics: Grand Challenges
Moderator: D.W. Grainger, University of Utah, J. Shumaker-Parry, University of Utah

8:00am **BI+AS+BM+MS-WeM1 Design of Antibody Array-Based Sensors for Disease Proteomics: Grand Challenges**, ***C. Wingren***, Lund University, Sweden **INVITED**

Antibody-based microarray is a new proteomic methodology setting a novel standard for analysing complex, non-fractionated proteomes. The first generation of antibody micro- and nanoarrays has already demonstrated its potential for generating detailed protein expression profiles, or protein maps, of human body fluids in health and disease, paving the way for new discoveries within the field of disease proteomics. The process of designing highly miniaturized, high-density and high-performing antibody array set-ups have, however, proven to be challenging. In this presentation, the key technological challenges that must be resolved in a cross-disciplinary manner before true global proteome analysis can be performed using antibody array-based sensors will be presented and discussed.

In this context, we have successfully designed a set of state-of-the-art recombinant antibody array technology platforms for high-throughput proteomics. In more detail, we use human recombinant single-chain Fv (scFv) antibody fragments, microarray adapted by molecular design as probes, displaying an outstanding on-chip functionality and stability. Uniquely, the platforms allows us to target both water-soluble as well as membrane proteins in a highly multiplexed and sensitive (pM to fM range) manner in complete, i.e. non-fractionated, directly labeled complex proteomes. Platforms compatible with a wide range of proteomes, including serum, plasma, urine, cell lysates, tissue extracts, intact cells etc, have been successfully designed. In addition, the first steps towards implementing label-free sensing (MS, MS -MS and SPRi) as well as designing self-addressable microarrays and miniaturized attovial-based nanoarrays as well as planar nanoarrays have been taken, clearly expanding the repertoire of technology platforms. The applicability of the platform(s) for differential high-content screening of clinical samples has been validated in a set of key applications within the field of oncoproteomics, autoimmunity, inflammatory diseases and allergy. The optimized antibody microarray technology platforms, as well as data from the screening analysis will be presented in context of the grand challenges the field experiences.

8:40am **BI+AS+BM+MS-WeM3 Development, Validation and Application of Q-Plex Array Technology**, ***M. Groll***, Quansys Biosciences Quansys Biosciences **INVITED**

The Quansys Q-Plex (multiplex ELISA) Array is a fully quantitative ELISA-based test where up to 25 distinct capture antibodies have been absorbed to each well of a 96-well plate in a defined array. This array is composed of 20 nanoliter spots with 350µm diameters and a pitch of 650µm between spots. Each spot represents a different distinct capture antibody population.

Using less than 30 µl of sample, up to 84 different samples can be assayed for all 25 unique analytes in less than 2.5 hours. Sensitivity is system dependent and typically ranges between 30 pg/ml to less than 1 pg/ml. All of the antibodies used in the Q-Plex arrays have been subject to a rigorous and comprehensive cross reactivity protocol and verified to be non-cross reactive with any other system on the array. Detection of this array is performed using the Quansys Q-View Imaging System. The image is then auto-processed using Quansys Q-View Software and concentrations for each analyte are output for the sample.

9:20am **BI+AS+BM+MS-WeM5 Drop on Demand Ink Jet Methods for Development and Manufacturing of Array Based Sensors and Diagnostics**, ***T.C. Tisone***, *A.V. Lemmo*, BioDot Inc.

The development and manufacturing of array based formats requires the transfer of biomarker reagents to a carrier substrate which forms the basis of a sensor for executing a multiplexed assay for research and diagnostics applications. The typical volume range for these types of assays is in the range of 100 pL up to 1000 nL: which lies in the range of commercial drop on demand piezoelectric and solenoid drop on demand dispensers. This presentation will discuss aspects of the physics and chemistry of successful applications of drop on demand methods to provide quantitative and high throughput reagent transfer to sensor substrates suitable for both Development and Manufacturing. Issues of drop formation, drop/substrate interactions and reagent/substrate interactions will be discussed. The agenda is to understand what role dispensing plays in the assay function.

10:40am **BI+AS+BM+MS-WeM9 New Molecular Strategies to Suppress Noise and Amplify Signal in Protein and DNA Microarrays**, ***A. Chilkoti***, Duke University **INVITED**

This talk will highlight recent work from my laboratory that addresses new interfacial technologies to suppress noise (N) and amplify signal (S) leading to heterogeneous assays with extraordinarily high S/N. In the first demonstration, I will focus on the adventitious adsorption of proteins as the primary factor that controls the limit-of-detection (LOD) of protein microarrays and limits the measurement of analytes from complex mixtures such as serum or blood. I will show data on a new protein microarray assay where background adsorption is effectively eliminated through the use of a protein-resistant –nonfouling– polymer brush. These "zero background" protein microarrays were successfully used to quantify protein analytes in serum with femtomolar LOD and a dynamic range of six orders of magnitude of analyte concentration. These LODs are 100-fold lower when compared to the same protein microarrays spotted on a conventional polymer substrate that displays high binding capacity but significant adventitious protein adsorption. This study also provided the first demonstration of the interrogation of an analyte directly from undiluted, whole blood by a protein microarray with a LOD of ~15 fM. Next, I will summarize recent work in my laboratory on the development of a new isothermal fluorescence signal amplification and detection scheme that exploits the ability of terminal deoxynucleotidyl transferase (TdTase) to add up to 100 fluorescent nucleotides to the end of a short DNA tag with an exposed 3'-OH. I will show how DNA microarrays that are printed on the nonfouling polymer brush exhibit low background signal, yet allow on-chip fluorescence signal amplification, leading to DNA microarrays that exhibit a sub-picomolar LOD, which appears to be the lowest LOD reported for DNA microarrays, to date.

11:20am **BI+AS+BM+MS-WeM11 SwitchDNA Biosensors for the Label-Free Detection and Sizing of Protein Targets on a Chip**, ***U. Rant***, *W. Kaiser, J. Knezevic, E. Pringsheim, M. Maruyama, P. Hampel,* Technische Universitaet Munich, Germany, *K. Arinaga,* Fujitsu Laboratories Ltd., Japan, *G. Abstreiter*, Technische Universitaet Munich, Germany

We introduce a chip-compatible scheme for the label-free detection of proteins in real-time that is based on the electrically driven conformation-switching of DNA oligonucleotides on metal surfaces. The switching behavior is a sensitive indicator for the specific recognition of IgG antibodies and antibody-fragments, which can be detected in quantities of less than 1 amol on the sensor surface. Moreover, we show how the dynamics of the induced molecular motion can be monitored by measuring the high-frequency switching response as well as by time-resolved fluorescence measurements. When proteins bind to the layer, the increase in hydrodynamic drag slows the switching dynamics, which allows us to determine the size of the captured proteins. We demonstrate the identification of different antibody fragments by means of their kinetic fingerprint. The switchDNA method represents a generic approach to simultaneously detect and size target molecules using a single analytical platform.

11:40am **BI+AS+BM+MS-WeM12 Nanomechanical Readout of DNA Microarrays**, *S. Husale,* Rowland Institute at Harvard University, ***H.H.J. Persson***, Stanford University, *O. Sahin*, Rowland Institute at Harvard University

DNA microarrays have enabled high throughput analysis of gene-expression and genotyping. However, they still suffer from limited dynamic range and rely heavily on enzymatic manipulations and amplification to create detectable signals. Here we present application of a novel nanomechanical detection method to microarray analysis that may circumvent these disadvantages. It is based upon a modified atomic force microscope (AFM) that can map mechanical properties of surfaces at high speed and spatial resolution. Mechanical measurements can reliably discriminate single and double stranded DNA on a surface. Automated image analysis reveals hybridized molecules with single molecule precision, thus providing a digital measure of hybridization. This method can detect a broad range of target concentrations with a limit of detection in the low attomolar concentration range without any labeling, enzymatic manipulations, and amplification. We demonstrate the performance of this technique by measuring differential expressions of miRNAs in tumor samples, which has been shown to help discriminate tissue origins of metastatic tumors.

Manufacturing Science and Technology
Room: C3 - Session MS+GR+MI-ThM

Manufacturing Issues for Beyond CMOS Nanoelectronics

Moderator: R.E. Geer, University at Albany

8:40am **MS+GR+MI-ThM3 Spin-Polarized Electrons in Silicon**, ***B. Huang***, *I. Appelbaum*, University of Maryland **INVITED**

In this talk, I will show how ballistic hot electron transport can be used for spin injection and detection in silicon. With this technique, we measure long conduction electron spin lifetimes which enable spin transport in silicon over long distances (up to 2mm). I will also discuss our investigations of spin dephasing and spin precession in oblique magnetic fields, and show how we realized spin precession control with an electric field.

9:20am **MS+GR+MI-ThM5 Methods for Characterizing Variations in Excitation Mode Frequency and Linewidth in Spin Transfer Nanocontact Oscillators**, ***M.R. Pufall***, *W.H. Rippard*, National Institute of Standards and Technology **INVITED**

Resonance probing of magnetoelectronic nanostructures with AC spin torque promises to provide a new means to understand their magnetic behavior, and their interaction with spin-polarized currents. An AC current produces an AC spin polarized current, which in turn produces a time-varying torque. By varying the frequency of the current, the resonance spectrum of the structure can be investigated. By this method, the ferromagnetic resonance mode of metallic and tunnel junction nanopillars has been investigated, and in nanocontact structures, enables probing the ferromagnetic resonance and damping of continuous films at unprecedented length scales.

However, for this tool become the more generally useful, the details of the signals produced by AC spin torque must be better understood. Beyond the ferromagnetic resonance mode, other responses are observed that have not been predicted; in nanocontacts, due to the unbounded geometry, prediction of modes beyond the ferromagnetic resonance in even more difficult. Furthermore, the shape of the ferromagnetic resonance line itself can vary in a complicated way, depending on the sample geometry and materials. As a step towards the goal of developing a robust tool that gives quantitative information about nanocontact spin transfer oscillators, I will present AC spin torque measurements from a variety of field geometries, and of materials with either in- or out-of-plane anisotropy, describing the basic behavior observed in each case. Then, I will compare different methods of ferromagnetic resonance detection (frequency-swept linewidth, field swept linewidth, field or microwave modulation) and discuss the challenges associated with interpreting these results to obtain the damping constant and the zero-field field-swept linewidth.

10:40am **MS+GR+MI-ThM9 Large Area, Continuous Single- and Few- Layer Graphene Films on Insulating Substrates**, ***J. Kong***, Massachusetts Institute of Technology **INVITED**

Graphene has exceptional electronic, thermal and mechanical properties. For the realization of graphene-related applications, it is necessary to develop reliable and low cost fabrication methods of graphene-based structures, ideally on any substrates. In this talk I will present our method of fabricating large area (~cm^2) films of single- to few-layer graphene and transferring the films to arbitrary substrates. The graphene films are synthesized by ambient pressure Chemical Vapor Deposition, consist of regions of 1 to ~10 graphene layers and have an average thickness of 2-3 nm. The structure of the graphene films are characterized with various methods, such as atomic force microscope, transmission electron microscope, scanning tunneling microscope and Raman. Detailed understanding in the growth mechanism provides guidance for improving the quality of the graphene films. The method presented in this work can potentially be scaled to industrial production of graphene films, for applications such as ultra-thin conductive and transparent electrodes, or devices and interconnect for integrated circuits.

11:20am **MS+GR+MI-ThM11 Graphene Nanoelectronics for Post-CMOS Logic Switches**, ***C.Y. Sung***, IBM T.J. Watson Research Center **INVITED**

Electron charge has been the computational state variable for decades. However, a new switch is urgently needed because scaling may fail to keep providing performance-cost benefits. We report the scaling limits and graphene research in monolayer synthesis, transistor engineering and new state variable logic switches. We demonstrate graphene nanoelectronics feasibility by monolayer-control wafer-scale synthesis, high performance device fabrication, bandgap engineering, for low-power, low noise performance and process integration. Computation with less power requires switches with alternative state variables. Graphene, with many desirable properties, emerge as a promising post-CMOS logic candidate.

Thursday Afternoon, November 12, 2009

BioMEMS Focus Topic
Room: A8 - Session BM+MN+MS+TF+BI-ThA

Advances in Microfluidics for BioMEMS

Moderator: G.W. Rubloff, University of Maryland

2:00pm **BM+MN+MS+TF+BI-ThA1 Advances towards Programmable Matter**, ***D. Erickson***, Cornell University **INVITED**

A dichotomy exists between the bottom-up self-assembly paradigm used to create regular structures at the nanoscale, and top-down approaches used to fabricate arbitrary structures serially at larger scales. The former of these enables rapid, highly parallel assembly but lacks critically important features of the latter such as the ability to arbitrarily direct the assembly location and perform error correction. We and our collaborators have recently proposed an alternative approach which combines these two based on dynamically programmable self-assembling materials, or ***programmable matter***. The uniqueness of our approach is that it uses dynamically-switchable affinities between assembling components facilitating the assembly of irregular structures. In this talk I present an overview of our approach and detail some of the analytical and experimental advances towards a programmable matter system we have recently made. These include: the development of a multi-chamber microfluidic chip for improved far-field assembly, the demonstration of near-field inter-tile affinity switching using a thermorheological assembly fluid and the ability to enhance assembly in three dimensions using unique fluid-structure interactions.

2:40pm **BM+MN+MS+TF+BI-ThA3 A Multilayered Microfluidic System with Buried Channels and Cell Compartmentalization for Engineering Heterogeneous Neural Networks**, *C. James, A. Greene,* ***A. Schiess****, G. Bachand,* Sandia National Laboratories*, M. Romero-Ortega*, University of Texas at Arlington

Current technology for engineering *in vitro* neural networks utilizes cell guidance cues that yield only temporary networks (< 1 month) as the cells rapidly diverge from their designed guidance cues during development of the culture. In addition, these engineered networks are typically comprised of homogeneous populations of neurons, thus the lack of multiple neuron types produces oversimplified networks that do not adequately represent in vivo networks. In addition, effective control over synaptic connections between different populations of neurons has not been demonstrated. Here, we describe a novel hybrid technology of multi-layered microfluidics with compartmentalized chambers containing multiple neuron types for engineering robust and complex neural networks with high resolution organization of synaptic connections. The device contains a first level of microfluidic channels etched 1-2 microns into the base glass substrate. These channels are fabricated with a novel process using a silicon nitride mask for hydrofluoric acid undercut etching to create buried microfluidic channels for robust containment and guidance of neurons. After the etching process, photoresist liftoff is performed to selectively adsorb poly-L-lysine (PLL) within the buried channels for improved neuron attachment and outgrowth at pre-defined locations. Polarity control of neurons is provided through a continuous set of guidance cues to promote axon development, while interrupted sets of guidance cues promote dendrite development. Current results show that axons and dendrites are positioned at predefined locations with a >65% accuracy. A second level of microfluidic channels and large (~mm) cell chambers are fabricated in polydimethylsiloxane (PDMS) from two-level SU-8 master molds. The base glass substrate and the PDMS substrate are aligned and bonded to create interconnects between channels in both substrates. These interconnects provide interaction regions for the development of synapses between growing neurites from cells in different chambers. We are currently applying this technology to engineer corticostriatal networks, an important region of the brain responsible for integrating multiple informational inputs crucial to complex decision-making in higher mammals. Specifically, we are using patch-clamp electrophysiology to track the development of synaptic memory in the form of long-term depression and potentiation (LTD/LTP) in these engineered networks.

3:00pm **BM+MN+MS+TF+BI-ThA4 Vesicle Production on a Microfluidic Platform using pH Sensitive Block Copolymers**, ***L.E. Brown***, The University of Sheffield, UK*, S.L. McArthur,* Swinburne University of Technology, Australia*, G. Battaglia, P.C. Wright*, The University of Sheffield, UK

The development of pH sensitive, biocompatible block copolymer vesicles has enabled the intracellular delivery of water soluble drugs and proteins. Improving the encapsulation efficiency of the vesicles is now a critical parameter. Transferring the production method to a microfluidic device creates the potential to vary the encapsulation conditions and improve this efficiency. In this work, a flow focussing microfluidic device is used. The self assembly of PMPC-b-PDPA block copolymer vesicles is induced within the device by changing the pH of the flows within the microchannels. The use of pH shift eliminates the need for organic solvents currently required for glass capillary production methods. This enables the biocompatibility of the block copolymers to be maintained, an essential factor for their application as molecular delivery vehicles.

The flow focussing microfluidic device was produced through standard soft lithography techniques. A three-channel flow system is used with the copolymer in solution at pH6 in the central channel and aqueous buffered solutions flowing in the channels either side. The laminar flow conditions within the microfluidic device result in a pH gradient at the interfaces where the three channels meet and where the block copolymers self-assemble into vesicles. These vesicle formation processes have been imaged using confocal microscopy via FRET with a block copolymer containing both rhodamine and fluorescein isothiocyanate groups. Dynamic light scattering and TEM were used to confirm vesicle formation.

With 50nm to 250nm vesicles continuously being produced within the device it was then possible to investigate whether higher encapsulation efficiencies can be achieved using the microfluidic device. The protein myoglobin was introduced through the central channel along with the copolymer. Spectrophotometric analysis indicated the overall the efficiency of the encapsulation process within the device is not a significant improvement on the standard bulk methods currently used, involving sonication of the vesicle solution containing the molecule to be encapsulated. Despite this, the continuous nature of microfluidic devices, as well as the lack of organic solvents being used in the production process indicates that the development of these devices offers a viable alternative production method for polymer vesicles that may enable the increases in encapsulation efficiency to be achieved. Work is ongoing to achieve this using the same pH shift mechanism within a glass capillary microfluidic device.

3:40pm **BM+MN+MS+TF+BI-ThA6 Integration of a Microfluidic Flow Cell Array with SPR Microscopy for In Situ Microarray Formation and Biomolecule Interaction Analysis**, *J. Liu, M. Eddings,* University of Utah*, A. Miles,* Wasatch Microfluidics*, B. Gale,* ***J. Shumaker-Parry***, University of Utah

Analysis of biomolecule interactions based on surface plasmon resonance (SPR) microscopy provides a label-free approach to monitoring arrays of biomolecule interactions in real time. Typically the microarray sensing surface for these measurements is prepared ex situ and a single or few channel flow cell is used for the biomolecule interaction studies. The multiplexing nature then is derived from the microarray and the number of samples that can be run simultaneously is rather limited, diminishing the potential application for assays requiring a high-throughput approach due to a large number of samples. One example of this is the need to monitor for anti-drug antibodies from a large pool of patient samples during clinical trials of biotherapeutics. We demonstrate the capability of a multi-channel microfluidic flow cell array (MFCA) to expand the throughput capability when integrated with SPR microscopy. In addition, the MFCA provides an in situ approach to array fabrication that allows full characterization of the biomolecule immobilization process. We use the MFCA for delivery of sample solutions with continuous flow in 48 channels in parallel for rapid microarray creation and binding analysis while using SPR microscopy for real-time monitoring of these processes. Label-free measurement of antibody-antibody interactions demonstrates the capabilities of the integrated MFCA-SPR microscopy system and establishes the first steps of the development of a high-throughput, label-free immunogenicity assay. We demonstrate a limit of detection (LOD) of ~ 80 ng/ml for the particular antibody pair we studied. This LOD is ~6 times lower than the industry recommended immunogenicity assay detection limit. The high-throughput nature of the integrated system allows a large number of replicate experiments, including control experiments, to be performed simultaneously on the same sensor surface in a short time. The integrated system also will be applicable for more general high-throughput protein-array based analysis.

4:20pm **BM+MN+MS+TF+BI-ThA8 Nanochannel Stretching of Nucleic Acids: Towards Epigenetic Analysis**, *D.E. Streng, S.-F. Lim, A. Karpusenka, J. Pan, J.A. Hook,* ***R. Riehn***, NC State University

Nanochannels with a diameter of about 100nm2 are a novel method for stretching DNA for genomic investigations. Such devices are implemented through standard nanolithography in fused silica. The elongation of DNA

results from an interplay of steric and entropic effects. Previous applications of nanochannel stretching included sizing, restriction mapping, and observation of transcription factor binding.

We show here that nanochannels can also be used to map the site-specific epigenetic state of DNA. In particular, we show here that the concept by nanoconfinement can be extended to chromatin, or DNA complexed to histones, and that the stretching is within the range expected from the de Gennes theory. We also demonstrate that the location-resolved cytidine methylation state of DNA can be mapped by specific fluorescent labeling. We will discuss the basic operation of these technique, and the application to artificial substrates with predefined epigenetic marks.

4:40pm **BM+MN+MS+TF+BI-ThA9 Microfluidic Models of Endothelial Cell Sprouting in Response to Biomechanical and Biochemical Microenvironments**, ***A.M. Shamloo***, *S.C. Heilshorn*, Stanford University

A novel microfluidic device was designed in order to generate stable, quantifiable concentration gradients of biomolecules in a cell culture chamber for 2-D and 3-D studies of shear-sensitive cell types such as endothelial cells. Endothelial cells form the inner lining of blood vessels and initiate a critical step in angiogenesis (the sprouting of new blood vessels) during wound healing and cancerous tumor growth. Therefore, a deeper understanding of the critical biomechanical and biochemical factors regulating endothelial cell sprouting can lead to improved clinical therapies for a multitude of diseases. Concentration distribution of soluble growth factors inside the microfluidic cell culture chamber was determined by simulation and experiment, and the stability of the gradient was verified over multiple hours. This device allows independent tuning of the matrix rigidity, the growth factor absolute concentration, and the growth factor concentration gradient steepness within a single experimental platform. Sprout formation of dermal microvascular endothelial cells was studied within collagen gels of varying density (0.3 - 2.7 mg/mL, corresponding to shear moduli of 8 – 800 Pa) that contained stable gradients of soluble vascular endothelial growth factor (VEGF). These experiments revealed that endothelial sprouting into multi-cellular, capillary-like structures is optimized at an intermediate collagen matrix density (G'~100 Pa). At lower matrix densities, cells were more likely to lose their coordinated motion and migrate as individual cells through the matrix; while at higher matrix densities, the cells formed broad cell clusters that rarely elongated into a sprout. Sprout thickness directly correlated with matrix rigidity, with thicker and less frequent sprouts present in gels with the highest shear moduli. Intriguingly, our 3D experiments also found that endothelial sprouts alter their sensitivity to VEGF depending on the matrix density, suggesting a complex interplay between biochemical and biomechanical factors. As matrix stiffness increases, steeper VEGF gradients and higher VEGF absolute concentrations are required to induce directional sprouting. In more compliant gels, endothelial sprouts that originally misaligned were able to turn and properly reorient parallel to the VEGF gradient; however, this turning phenomenon was only rarely observed in stiffer gels. These results demonstrate that matrix stiffness is an effective factor in stabilization and orientation of endothelial cells during sprouting and suggests new anti-angiogenic strategies for potential cancer treatment as well as pro-angiogenic strategies for regenerative medicine scaffolds.

5:00pm **BM+MN+MS+TF+BI-ThA10 Plasma Polymerisation of PDMS for Microfluidic Applications**, ***S. Forster***, *A.G. Pereira-Medrano, G. Battaglia, P.C. Wright,* University of Sheffield, UK, *S.L. McArthur*, Swinburne University of Technology, Australia

Polydimethylsiloxane (PDMS) has become the most popular material choice for a wide range of microfluidic bioengineering applications, including proteomics, protein separations and drug discovery and development. The reasons its popularity lie mainly in its highly advantageous fabrication requirements when compared to traditional materials such as glass and silicon. However, PDMS has some fundamental drawbacks, namely a lack of functionality present at the surface, high protein fouling and an inability to retain stable surface modification due to its motile hydrophobic monomer. These factors can lead to the loss of specificity and sensitivity in many bioassays. Due to this reason much work has been completed looking into surface modification of PDMS for such applications. Here an alternative method of stable surface modification of PDMS for many microfluidic applications through enhanced curing conditions and plasma polymerisation is shown. Stable and functional surface coatings have been achieved on bulk PDMS and within microfluidic channels. Bulk surfaces were characterised using a combination of XPS and ToF-SIMS, while coated micro-channels were tested using confocal microscopy and various assays. This methodology has been used in many applications and one area where it has proven extremely useful is in microfluidic proteomics where surface properties are of paramount importance due to the inherently small volumes and quantities associated with biological samples. Firstly, plasma polymer coated PDMS micro-channels were utilised for on-chip IEF protein separations (i.e. separating proteins bases on charge) and showed reduced electrosmotic flow (EOF) and protein adsorption within the device. Secondly, a µIMER (micro-immobilised enzyme reactor) was produced using plasma polymer coated PDMS devices. The µIMER was then used in 'shotgun' protein digestion applications in conjunction with Mass Spectrometry where it was shown to have numerous advantages over untreated PDMS devices, as well as comparing favorably to published work on other µIMER systems. The device was used to digest single and multiple protein samples as well as complex membrane protein samples. Finally, successful covalent bonding of plasma polymer coated devices has led to the completion of polymer vesicle immobilisation within a microfluidic channel. Initial work looking at the immobilisation of polymer vesicles with an encapsulated digestive enzyme has shown to increase proteomic digestion efficiency. This success opens up the possibility of translating this technique into many potential microfluidic applications through the extensive versatility of encapsulation within polymer vesicles.

Manufacturing Science and Technology
Room: C3 - Session MS-ThA

Manufacturing Issues in Nanoelectronics, PV and SSL
Moderator: C.Y. Sung, IBM Research Center

2:00pm **MS-ThA1 Nanoelectrical and Nanomechanical Interconnect and Device Metrology for CMOS Extension**, ***R.E. Geer***, *C.H. Chong, Y. Wang*, University at Albany **INVITED**

So-called 'equivalent scaling' which is dominating the extension of CMOS from the 'pure' scaling regime places inordinate demands on interconnect performance both for conventional CMOS switch configurations as well as alternate switch materials (III-V, carbon-based). As a result, substantial advances are required in fundamental metrology measurements of thin film electrical continuity in conventional intra-core and core-core interconnects as well as innovative approaches for electrical and mechanical interconnect metrology for alternate CMOS material sets. Here, we present nanoscale electrical continuity profiling of ultra-thin barrier and Cu films for conventional interconnects as well as nanoscale electrical metrology of alternate material (graphene-based) interconnects. For the former, it is shown that sub 2-nm films, although electrical conductive, show local reduction in electrical continuity that correlate to line-edge-roughness in patterned interconnect test structures. In contrast, alternate interconnect materials (e.g. single-layer graphene) are shown to exhibit electrical uniformity although local defectivity and electrostatic doping (for the case of interconnect applications) must be sufficiently controlled for use in conventional CMOS geometries.

2:40pm **MS-ThA3 New Mechanism for Optically Stimulated Point Defect Control In Ultra-Shallow Junction Formation**, *P. Gorai, Y. Kondratenko,* ***E.G. Seebauer***, University of Illinois at Urbana-Champaign

Formation of pn junctions in advanced Si-based transistors employs rapid thermal annealing (RTA) after ion-implantation in order to increase the activation of dopants. There has long been suspicion that the strong lamp illumination in RTA equipment may nonthermally influence the diffusion of dopants. The present work describes the evidence for a photostimulated diffusion mechanism based on electrostatic coupling between interface and Si bulk. Photostimulated effects on diffusion of boron were studied in ion implanted crystalline silicon samples. Low intensity illumination (2 W/cm^2) was used for nonthermal photostimulation during soak annealing using with resistive heating of the substrate. This experimental design allowed decoupling of heating and illumination. The samples were annealed at different temperatures and dopant diffusion and activation data was compared between experiments with and without illumination. Experimental data in conjunction with continuum simulations showed that light interacts with the charged defects at Si-SiO_2 interface and modulates the electrical field arising from near-surface band bending. The effects of this modulation exhibited profound effect on diffusion profile evolution near the surface and in the bulk. Simulations results were further employed to elucidate underlying physical mechanism of this effect.

3:00pm **MS-ThA4 Thickness/Composition Metrology of Ultra-thin Lanthanum Oxide Cap Layer for CMOS Metal Gate Work Function Tuning**, ***C.C. Wang***, *Y. Cao, G. Liu, X. Tang, Y. Uritsky, S. Gandikota*, Applied Materials Inc.

Beyond the 45 nm node CMOS application, metal gate and high-k dielectric are used and many new thin film materials are developed. In order to reduce the threshold voltage of the CMOS gate, the matching of the metal gate work function with the silicon band position is important. For NMOS work

function tuning, lanthanum oxide (LaO_x) thin film is used. Device performance demands the use of less than 10 Å thick LaO_x and the stringent control of thickness and composition uniformity (1 to 2% 1σ) on 300 mm wafers. However, metrology of this new material is very difficult. First the LaO_x cap layer is so thin, only ellipsometry, X-ray photoelectron spectroscopy (XPS) and X-ray fluorescence spectroscopy (XRF) have the required sensitivity. Second, the composition of LaO_x changes with air exposure time due to its reaction with ambient moisture; such behavior renders ellipsometey and XPS ineffective, because the composition result and the thickness result from both techniques are strongly correlated in the case of ultra-thin film measurements. This leaves XRF to be the only candidate for both composition and thickness measurements. The main advantage of XRF is that the X-ray signals from the sample are proportional to the surface doses (atoms/cm^3) of the elements in the ultra-thin film; hence, it is simple to calibrate the tool and develop measurement recipes. To satisfy the new metrology needs, wavelength dispersive XRF (WD-XRF) and energy dispersive XRF (ED-XRF) techniques were developed.

For tool calibration and drift monitoring, a LaO_x thin film standard with known thickness, density and composition had to be prepared. Due to high reactivity of LaO_x in air, it was difficult to prepare a stable standard and to determine all its attributes. To overcome this problem, a 200 Å thick LaO_x thin film on silicon standard capped with a thin TiN layer was prepared, its thickness and density were measured by X-ray reflectivity and its composition was derived by the XRF measurement itself by an ingenuity method. Due to the good sensitivity of WD-XRF to both La and O signals, recipe was developed to monitor the composition of freshly deposited PVD LaO_x thin films and their change with ambient exposure time. The results showed that the LaO_x ultra-thin film deposited with lanthanum oxide target was more stable in air than those deposited with a La metal target. ED-XRF recipe was developed to monitor the LaOx thickness uniformity on 300 mm wafers. The main advantage of ED-XRF was its small X-ray spot size that afforded 3 mm edge exclusion measurements. Study showed ED-XRF had < 1% (1σ) precision with good throughput of < 60 seconds per data point.

3:40pm **MS-ThA6 Sidewall Image Transfer for Sub Lithographic Pitch Scaling for the 22nm CMOS Node & Beyond**, ***S. Kanakasabapathy***, IBM Research, *R.H. Kim,* Global Foundries, *A. Ko, A. Metz,* Tokyo Electron Limited, Japan, *T. Osabe,* Hitachi Technologies, Japan, *S. Schmitz, T. Standaert*, IBM Systems and Technology

Critical Dimension (CD) Scaling and Pitch Scaling for the past several decades have sustained the Microelectronics Industry's march along the Moore 's law. Wavelength, Numerical Aperture & Immersion assisted index scaling have made possible such pitch scaling in a fashion relatively transparent to Etch. However with the technical and manufacturing challenges faced by Extreme Ultraviolet (EUV) wavelength scaling, Etch/Integration assisted Pitch Scaling is being explored. Ground rule challenges have grown exponentially since Sidewall Image Transfer (SIT) was proposed as such a technique for pitch halving.

We present herein SIT challenges for obtaining sub 80 nm pitches for Line space levels compatible with Front End of Line applications. Line Edge Roughness (LER) and Line width Roughness (LWR) measurements for these integration schemes will be presented with options to mitigate them.

4:00pm **MS-ThA7 Wafer-level Process Sampling, Metrology and Testing for MEMS and Solar Photovoltaic Applications**, ***V. Ngo***, FEI Company

As MEMS (Micro-Electro-Mechanical Systems) devices further proliferate many industrial and consumer products, the need for fast characterization becomes critical to production process support and time-to-answer in failure analysis. For example, a Dual-Beam can be used to characterize the MEMS fabrication processes by cross-sectioning the area of interest using a focused ion beam and imaging the uncovered feature of interest for metrology and process evaluation using a scanning electron beam or via the ion beam itself. The ion-beam can also be used to weld and cross-section fragile suspended features without additional process steps such as resist backfilling. Most MEMS components, however, are very large on the order of 10s-100s microns, which can lead to long sample preparation times. Furthermore, localized gas chemistry injection on the micron-level at the point of beam-sample interaction further enhance milling rate to support wafer-level multi-site sampling for near-production process support. FA work in MEMS packaging and TSV development for MEMS-CMOS integration also have demonstrated benefits from the gas-enhanced FIB de-processing.

In addition to sample preparation throughput, it is possible to use a micromanipulator probe to remove specific components of the device for characterization. This work also demonstrates that A FIB system configured with such an in-situ sample lift-out probe can remove specific components at their attachment points and lifting them out to characterize their etch profile, rather than attempting to ion mill a large area away to enable access. Both gas-enhanced and lift-out approaches can significantly reduce the amount of time required for a cross-section of the device, and improve the quality of data obtained.

4:20pm **MS-ThA8 Establish Degradation Rates of Photovoltaic Modules and Systems Trough Comprehensive Electrical and Mechanical Analysis**, ***A. Leyte-Vidal***, *K. Davis, W. Wilson, R. Reedy, N. Hickman,* Florida Solar Energy Center, *S. Kurtz*, National Renewable Energy Laboratory

Performance degradation of photovoltaic modules and systems follows a progression that is dependent on multiple factors. Many of the mechanisms responsible for degradation are difficult to simulate in a laboratory setting. While accelerated aging tests are a valuable tool in evaluating photovoltaic components and systems, long-term monitoring of systems installed in the field is the true test of reliability. A comparison of the original and weathered power output of several different photovoltaic technologies dating back to 1998 is presented here, along with an analytical description of the degradation and weathering effects responsible for reduced power production over extended periods of time. Experimental data has been collected on diverse generations of photovoltaic modules installed throughout the state of Florida, where the systems have been subjected to long-term exposure in a hot, humid climate. Some of the module degradation mechanisms may be attributed to optoelectronic effects, while others are more mechanical in nature (e.g. encapsulation and delamination issues). The effect of performance degradation on the system's economics and life-cycle energy costs has been presented in order to better quantify the impact of the different degradation mechanisms. While working to reduce the initial degradation effects is of vital importance and has received considerable interest in the past, a better understanding of the long-term degradation mechanisms inherent in this technology is also fundamental in the effort to improve the reliability of photovoltaic modules and systems.

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