Thin Films
Room 303C - Session TF-FrM

In-Situ/Ex-Situ & Real-Time Monitoring

Moderator: C.H. Stoessel, Consultant

8:20am **TF-FrM1 Real-time and Spectroscopic Second Harmonic Generation as a Tool to Probe Surface Processes during Amorphous Silicon Film Growth**, ***I.M.P. Aarts***, *J.J.H. Gielis, C.M. Leewis, M.C.M. van de Sanden, W.M.M. Kessels,* Eindhoven University of Technology, The Netherlands

We have explored the capability of second harmonic generation (SHG) to probe surface and interface processes during the growth of amorphous silicon-based thin films such as hydrogenated amorphous silicon (a-Si:H). The a-Si:H thin films are deposited under ultrahigh vacuum conditions by means of a hot wire source on fused silica substrates. Real-time spectroscopic ellipsometry has been used to monitor the thickness evolution and the optical properties of the a-Si:H films. SHG has been observed during in situ experiments on as-deposited films for various input and output polarizations configurations. Although SHG is surface and interface specific for isotropic media such as a-Si:H, interpretation in the present case is complicated because not only interference effects have to be taken into account but also the macroscopic origin of the SHG is not straightforward to determine. The experimental data has therefore been analyzed with a complete optical model that takes into account interference effects of both the fundamental and the SHG radiation as well as two nonlinear polarizing sheets in which the SHG signal is generated. These sheets are positioned at the surface and at the interface between the film and the substrate. Further understanding of the SHG signal has been obtained by performing spectroscopic SHG measurements, which showed two distinct resonance peaks at photon energies of 1.2 and 1.4 eV. After exposing the a-Si:H film to oxygen the SHG signal is quenched, indicating that the corresponding resonance peaks involve transitions with dangling bond states. In addition, real-time observation of the SHG signal during film growth and dosing experiments have already revealed that SHG is sensitive to nucleation and relaxation processes at the surface and/or interface region during and after growth of these silicon-based films.

8:40am **TF-FrM2 MultiChannel Mueller Matrix Ellipsometry for In-Situ and Real Time Analysis of Thin Films and Surfaces**, ***R.W. Collins***, *C. Chen, I. An, N.J. Podraza,* University of Toledo **INVITED**

A dual rotating compensator multichannel ellipsometer has been applied for real time Mueller matrix spectroscopy (2 to 5 eV) of anisotropic surfaces and thin films. The sequence of optical elements for this instrument is denoted PC@sub 1@SC@sub 2@A, where P, S, and A represent the polarizer, sample, and analyzer. C@sub 1@ and C@sub 2@ represent the first and second compensators that rotate at frequencies of 10 and 6 Hz, respectively, synchronized for a 5:3 ratio. At this ratio, the 2n@omega@ frequencies where n = 1, ..., 8, 10, 11, 13, and 16, and where @omega@/2@pi@ = 2 Hz, are present in the detected irradiance waveform for the most general sample Mueller matrix. The associated 25 experimental dc, cosine, and sine Fourier coefficients are determined for a given pixel of the photodiode array detector by integrating the waveform 36 times per optical cycle. Spectra in the 16 Mueller matrix elements can be determined from spectra in the 25 non-zero coefficients acquired in a single 250 ms optical cycle and from instrument calibration data obtained in advance. In high speed Mueller matrix measurements, this research has focused on weakly anisotropic surfaces and thin films that push the instrument to its limits. These include the (110) Si surface and thin films having an oriented columnar microstructure in which case the weak anisotropic optical response can be over-determined from analyses employing separate 2x2 blocks of the Mueller matrix. As a result, detection of anisotropy is definitive, and the co- and cross-polarization ellipsometric angles @psi@@sub pp@, @psi@@sub ps@, and @psi@@sub sp@ can be determined to within an accuracy of 1 part in 10@super 4@.

9:20am **TF-FrM4 Analysis of Ti and TiN Thin Film Nucleation, Coalescence, and Growth by Rotating Compensator Multichannel Ellipsometry**, ***C. Chen***, The Penn State University*; B. Hong,* Sungkyunkwan University, Korea*; P. Sunal, M.W. Horn, R. Messier,* The Penn State University*; R.W. Collins,* University of Toledo

Real time spectroscopic ellipsometry has been applied in the rotating-compensator configuration to characterize the nucleation, growth, and optical properties of titanium and titanium nitride thin films deposited by magnetron sputtering on silicon wafers with thermally-grown silicon oxide and nitride overlayers. The ellipsometer used in this study incorporates recent instrumentation advances for a wide spectral range (1.5 to 6.5 eV), including a dual Xe/D@sub 2@ source with an intervening iris for spectral flattening. The real time ellipsometric spectra collected throughout film deposition on the smooth wafer substrates are analyzed in terms of a transition from a one-layer to a two-layer optical model. This transition simulates the overall thin film structural evolution including nucleation, coalescence, and surface roughening during growth. In the initial stages, the spectra are particularly sensitive to smoothening that occurs during coalescence. In the case of Ti deposition on SiO@sub 2@ covered Si wafers, for example, this effect is maximized within a narrow deposition parameter window of Ar sputtering gas pressure and plasma power. In this case, 15-20 Å thick Ti clusters coalesce, yielding surface roughness only a monolayer in thickness after ~100 Å of Ti bulk layer deposition. The evolution of the optical properties of the Ti layer with cluster size during nucleation and with bulk thickness during growth provides additional information on the structure of the film and its electronic properties. Finally, insights from these studies can be used to direct the fabrication of alternating multilayer and nanocomposite films for hard-coating applications.

9:40am **TF-FrM5 Comparative Ellipsometric Study of Liquid Helium Thin Films on Au, Cs, HOPG and Rb Substrates**, ***T. McMillan***, *P. Taborek, J.E. Rutledge,* University California Irvine

We have developed a modulated null ellipsometer with sub-monolayer resolution to measure adsorbed liquid helium thin films at temperatures from 1.3 to 4.3 K. Measuring helium films pushes the limits of cryogenic ellipsometry due to helium's extremely small index of refraction. We have performed isotherms on substrates with a range of substrate potentials: Au is a representative strong substrate while Rb is an intermediate and Cs a weak substrate. These measurements will determine how the binding energy affects the superfluid transition. The ellipsometer allows us to explore effects that cannot be discerned solely from quartz crystal microbalance measurements, which are only sensitive to the normal fluid fraction of the film.

10:00am **TF-FrM6 Studies of Coupling and Ordering in Magnetic Thin Films with Polarized Neutron Reflectormetry**, ***S.G.E. te Velthuis***, Argonne National Laboratory **INVITED**

Traditionally neutron scattering has been an important tool for studying bulk magnetic materials. The success has been due to the high sensitivity of neutrons for magnetic moments, combined with their characteristics in relationship to wavelength and velocity. As the interest of the scientific community has shifted towards nanostructured materials, polarized neutron reflectivity (PNR) and scattering at grazing incidence have emerged as powerful methods for studying magnetic thin films. As will be illustrated by the presentation of several experiments, the depth dependent magnitude and orientation of the magnetization in a thin film can be determined with PNR. In the case of GaMnAs thin films knowledge about the magnitude of the magnetization provided understanding about the effect of temperature annealing in these films. Determination of the orientation of the magnetization of individual Fe layers in Fe/Cr[110] superlattices as was essential to the understanding of the magnetic coupling between the Fe layers spaced by Cr. Finally, PNR experiments performed with polarization analysis mapped out the evolution of individual magnetization vectors during the first order spin flop transition in a finite antiferromagnet, thereby confirming theoretical predictions about this magnetic phase transition.

10:40am **TF-FrM8 Real-time Optical Monitoring of Ammonia Decomposition Kinetics in InN Vapor Phase Epitaxy at Elevated Pressures**, ***N. Dietz***, *M. Strassburg, V. Woods,* Georgia State University

Understanding the decomposition kinetics of the chemical precursors involved in nucleation and thin film growth processes is crucial for controlling the growth process. The growth of emerging materials heterostructures such as InN and related alloys requires deposition methods operating at elevated vapor densities due to the high thermal decomposition pressure in these materials. High nitrogen pressure has been demonstrated to suppress thermal decomposition of InN, but has so far not been explored in chemical vapor deposition experiments. The extension of chemical vapor deposition (CVD) to elevated pressure opens an avenue for retaining stoichiometric single phase surface composition for materials that are characterized by large thermal decomposition pressures at optimum processing temperatures. In this contribution we present research results on the decomposition kinetics of ammonia in the laminar

flow regime of a high-pressure flow channel reactor. Real-time optical UV absorption and fluorescence spectroscopy has been applied to study the gas phase chemistry as function of flow, pressure and temperature. Data are presented for the optical methods of real-time process monitoring to analyze the decomposition process as well as the initial stages of InN heteroepitaxy.

11:00am **TF-FrM9 In-Situ Real-Time FT-IR Spectroscopy During APCVD: The Effect of B and P Dopants on SiO@sub 2@ Deposition**, *A. Effenberger, L.D. Flores,* ***J.E. Crowell,*** University of California, San Diego

In-situ FT-IR spectroscopy has been used to explore the chemical boundary layer (CBL) region formed during atmospheric pressure chemical vapor deposition. Infrared spectra are recorded in 22 second intervals while varying the precursor gas composition. Using TEOS and ozone precursors in combination with borates and/or phosphites, thin films of boro-, phospho-, or borophosphosilicate glass are deposited onto Si wafers at 725K. Gas phase intermediates containing SiOH and BOH functionalities have been observed, and their variation with chemical composition has been investigated. A partial least squares principle component analysis has been used to quantify the reactive chemical mixtures and to evaluate the effect of dopants on the chemical kinetics.

11:20am **TF-FrM10 Optimal Control on Composition and Optical Properties of Silicon Oxynitride Thin Films**, ***E.C. Samano***, *J. Camacho, R. Machorro,* CCMC-UNAM, Mexico

The desire to merge the most advantageous physical and chemical properties of both SiO@sub 2@ and Si@sub 3@N@sub 4@ in an optimum combination tailored to various applications in electrical, optical and optoelectronic thin films has pushed a continuous interest in the processing of SiO@sub x@N@sub y@ thin films. Inhomogeneous thin film filters have had an increasing importance in the industry as optical filters. Silicon oxynitride, SiO@sub x@N@sub y@, is a very interesting material for multiple applications, including graded refractive index films. The refractive index can be changed from pure silicon dioxide (1.47) to silicon nitride (2.4) by just varying the film composition. We report the growth of SiO@sub x@N@sub y@ films by reactive laser ablation using two different solid targets, Si@sub 3@N@sub 4@ and Si, in the ambient of N@sub 2@ and O@sub 2@ at various pressures, introduced separately in the growth chamber, as the film is monitored by real time ellipsometry. The oxidation rate in the films is studied for both targets. The composition of the films is in situ determined by AES and XPS. The evolution of the chemical bonding of the species in the film is done by FTIR. The SiO@sub x@N@sub y@ film stoichiometry, bonding character and optical properties are compared as a function of O@sub 2@ pressure while N@sub 2@ pressure is maintained fixed as either the Si@sub 3@N@sub 4@ or Si target is ablated.

11:40am **TF-FrM11 III Nitride-Based Optical Sensors Integrated with a TOF Mass Spectrometer for Aerosol Characterization.**, *D. Starikov, N. Medelci, R. Pillai,* Integrated Micro Sensors Inc.*; A. Bensaoula,* ***C. Joseph***, *Z. Mouffak,* University of Houston

Mass spectrometry is currently an essential aerosol characterization tool. For many important biochemical and biomedical applications it would be beneficial to perform optical characterization of the aerosol prior to mass spectrometry. Such characterization would include particle detection, evaluation of the particle size, shape, velocity, etc. Rugged multifunctional multi-wavelength solid-state optical sensors fabricated from III nitride based components have a miniature size and demonstrated high performance in detecting various important compounds. In this work integration of our miniature solid-state optical sensors based on III nitrides with a compact Time-of-Flight (TOF) mass spectrometer has been attempted. The sensors were tested ex situ by fluorescence, absorption, and scattering measurements performed on solutions of fluorescein, rhodamine, erythrosine, chlorophyll, pyrene, anthracene, alexa fluor, and red fluorescing microsphere fluorochromes. These measurements indicated that some of the compounds can be detected with a sensitivity as low as 4-5 ppm in a wide range of concentrations. After integration with an ORTOF mass spectrometer (Ionwerks Inc.), the sensors will be tested for detection and characterization of fluorescing and non-fluorescing aerosols. The sensitivity, dynamic, range, detection limits will be evaluated for various aerosol analytes.

Author Index

Bold page numbers indicate presenter