Electronic Materials and Processing Division
Room 608 - Session EM-FrM

In Situ Monitoring and Growth

Moderator: C.R. Abernathy, University of Florida

8:20am **EM-FrM1 Optical Techniques for Real-Time Measurement of Stress and Morphology During Thin Film Growth*, J.A. Floro,*** Sandia National Laboratories **INVITED**

Mechanisms for the generation and relaxation of stresses in thin films during deposition have been subject to extensive scientific research and technological tweaking. Stress evolution during film growth can be quite complex, and is best measured in real-time during the deposition process. The primary focus of this talk will be on the use of substrate curvature measurements, since this approach has the greatest degree of flexibility in its implementation. The use, limitations, and extensions of the Stoney equation, which relates substrate curvature to film stress, will be discussed. Methods for curvature measurement will be surveyed, with emphasis on a recently developed laser-deflection technique that is currently being applied to a wide range of thin film systems. I will present several instructive examples on the interpretation of real-time stress data obtained during the deposition of heteroepitaxial and polycrystalline thin films. Finally, a new spectroscopic light scattering technique will be introduced that provides real-time measurement of surface morphological evolution. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-ACO4-94AL85000.

9:00am **EM-FrM3 Insitu Surface Stress Measurements during Contact Reaction of Ultrathin Overlayers of Cobalt on Si(100) and Si(111)*, J.G. Nivison*,** *D.G. Waters, P.A. Bennett,* Arizona State University

We report insitu measurements of surface stress during the contact reaction of 10 Angstrom overlayers of cobalt on Si(111) and Si(100) using an optical deflection wafer curvature technique. The instrument features a feedback stabilized reference beam, which removes thermal drift to first order, allowing temperature dependent measurements. For Si(100), the surface stress during room temperature dosing is +0.2 N/m per Ml (tensile), saturating at 8Ml. Upon annealing, the stress increases monotonically to +0.6 N/m at 200C, which is attributed to a partially coherent CoSi2 overlayer. For Si(111), the stress during room temperature dosing is +0.6 N/m for the first Ml, changing to -4.5 N/m at 5Ml coverage. Upon annealing, the stress increases to +4.0 N/m at 450C, which is attributed to a fully coherent CoSi2 overlayer.

9:20am **EM-FrM4 Vibration Analysis of SiH@sub n@ Bending Modes on Hydrogenated Si(100) Surface Using Infrared Reflection Absorption Spectroscopy*, H. Noda,*** *T. Urisu,* Institute for Molecular Science, Japan*; M. Hiramatsu,* Meijo University, Japan

Detailed analyses have been successfully made for the SiH@sub n@ stretching vibration mode on hydrogenated Si(100) surface, which is of great scientific and technological interest.@footnote1@ However, concerning the bending vibration region, which gives important information about SiH@sub 2@ and SiH@sub 3@ species, very little work has been done. Recent developments of buried metal layer infrared reflection absorption spectroscopy (BML-IRRAS) have made the high-resolution vibration analysis of bending region easy. In this work, adsorption and desorption of hydrogen on Si(100) surfaces have been investigated by measuring BML-IRRAS covering a wide spectral range (800-2200 cm@super-1@). In both 3x1 and 1x1 phases observed with reflection high-energy electron diffraction (RHEED), a doublet peak (902 and 913 cm@super -1@) has been clearly observed and assigned to the SiH@sub 2@ scissors mode. The splitting of the peak is most likely due to the frequency difference of SiH@sub 2@ scissors vibration between single SiH@sub 2@ (ordered 3x1 units ; H-Si-Si-H H-Si-H H-Si-Si-H) and neighboring SiH@sub 2@ (disordered 3x1 units ; H-Si-Si-H H-Si-H H-Si-H H-Si-Si-H). Coverage and annealing temperature dependence of this doublet peak have also been investigated. @FootnoteText@ @footnote 1@ Y. J. Chabal and K. Raghavachari, Phys. Rev. Lett. 54 (1985) 1055.

9:40am **EM-FrM5 Assessment of Various Sensors for in-situ Monitoring and Control of MBE*, K.G. Eyink,*** Air Force Research Laboratory

Molecular Beam Epitaxy is a technique, which has been used to grow semiconductor thin film structures. In this process reactants fluxes are incident onto a substrate held at an elevated temperature. Typically the structure is grown by an appropriate shuttering sequence that is based on extensive pre-calibration. In this talk I am going to discuss the use of several sensors to monitor MBE in real-time. A discussion of the advantages and disadvantages of the various techniques will be given. The utilities of ellipsometry, desorption mass spectrometry, and atomic absorption for in-situ targeting of composition and growth rates will be given. As well as the advantage of integrating absorption band edge spectroscopy into substrate temperature control and its coupling to the various other sensors.

10:00am **EM-FrM6 Integrated Real-time SE, RDS and QMS to Characterize and Optimize OMCVD Growth*, K.A. Bell*,** *M. Ebert, S.D. Yoo, K. Flock, D.E. Aspnes,* North Carolina State University

We combine spectroscopic ellipsometry (SE) and reflectance-difference spectroscopy (RDS) in a single optical path with quadrupole mass spectrometry (QMS) on an OMCVD reactor to probe bulk and surface properties as well as ambient composition in real time during growth. This unique system enables us to characterize the complex parameter space of OMCVD and optimize growth conditions. Monitoring the optical response of the sample and gas phase species enables us to determine ideal growth parameters for our reactor and to characterize growth mechanisms. The optical spectrometer is a multichannel, parallel acquisition and processing system built around a commercial rotating-spindle OMCVD reactor and a custom photodiode array (PDA) detector that allows us to extract sample optical properties over a spectral range of 200 to 800 nm at a repetition rate of 2 Hz. Together with time-resolved QMS, we determine system-specific parameters necessary to calibrate temperature and characterize gas flow dynamics. The former has been done using reciprocal space analysis of SE data to extract sample critical point energies in real time with an accuracy of 0.5 meV corresponding to +/-1 degree in sample temperature. Monitoring the growth of GaP on Si(001) in real time, we observe a SE sensitivity to 0.1 Angstrom changes in thickness and a QMS sensitivity of 0.1% changes in ambient composition.

10:20am **EM-FrM7 Real-time Thickness and Compositional Control of Ga1-xInxP Growth using P-Polarized Reflectance*, V. Woods*,** *K. Ito, I. Lauko, N. Dietz,* North Carolina State University

Advances in the engineering and design of advanced electro-optical materials require sensors and control strategies that allow tight control over thickness and composition. In response to this demand, we developed p-polarized reflectance (PR) as a real time optical characterization technique, and demonstrate its sensitivity during heteroepitaxial GaP/GaInP growth under pulsed chemical beam epitaxy (PCBE) conditions. For closed loop control, we applied nonlinear control algorithms (based on nonlinear Kalman filtering) that utilizes the PR signals to control thickness and composition during heteroepitaxial growth of GaxIn1-xP on Si (001). A reduced order surface kinetics (ROSK) model has been formulated to describe the decomposition process of organometallic precursors and the time-dependency of the molar concentrations of the precursors fragments. These data are linked to compute the composition and thickness increase per time unit, utilizing the monitored PR signal for validation. This allows to establish feedback control algorithms, able to control both the growth rate and composition of GaxIn1-xP heterostructures.

10:40am **EM-FrM8 Migration-Enhanced Epitaxy of CuInSe@sub 2@*, B.J. Stanbery*,** *S. Kincal, S. Kim, O.D. Crisalle, T.J. Anderson,* University of Florida

We describe a novel rotating-disc MBE reactor that implements a Migration-Enhanced Epitaxy (MEE) process@footnote 1@ and its application to the growth of Copper Indium Selenide (CIS) on GaAs and ZnTe single-crystal substrates. MEE is a process variant of conventional MBE and one of a general class of Modulated-Flux Deposition (MFD) processes that are based on cyclic alternation between deposition and relaxation steps, and the separation of cationic and anionic species fluxes. All of these measures are intended to achieve long surface diffusion lengths and promote adatom/substrate equilibration. Our reactor is divided by carefully designed shielding into four nominally isolated zones: metals (Cu+In) deposition, cooling, chalcogen (Se,S) deposition and heating. The fixed substrate heater in the latter zone combined with the rotating-disc design results in cyclic heating and cooling of the substrates. The direction of substrate rotation can be reversed, enabling two different growth cycles. The cooling zone includes a source for dosing the substrates with dopants or surfactants. We will present the results of our molecular and thermal flux modeling and discuss their implications for surface growth kinetics in our reactor. We will also present XRD, AFM, Auger, SIMS and PL data characterizing our successful epitaxial growth by MEE of (001)CuInSe@sub 2@ on (001)GaAs and (001)ZnTe. Our data also shows phase segregation of

Cu@sub 2-X@Se in copper-rich epilayers and a relative loss of crystal quality in indium-rich epilayers. We will discuss the relevance of these results to our free-energy thermochemical defect model of the Cu-In-Se ternary system. We show that these data provide evidence of a transition from Stranski-Krastonov to Frank-Van de Merwe growth mode when the surface is dosed with small quantities of sodium fluoride during the initial stages of epitaxy on (001)ZnTe, supporting our thesis@footnote 2@ that sodium behaves as a surfactant in this material system. @FootnoteText@ @footnote 1@ Y. Horikoshi, M. Kawashima, and Y. Yamahuchi, Jap. J. Appl. Phys. 25, 1986, p. L868. @footnote 2@ B.J. Stanbery, C.-H. Chang, and T.J. Anderson, 11th International Conference on Ternary and Multinary Compounds, 1997, Inst. of Phys. Conf. Series 152, pp. 915-922.

11:00am **EM-FrM9 Boron Nitride Thin Films for High Temperature Multilayer Ceramic Capacitor Chips (MLC3's)***, **N. Badi**, D. Starikov, N. Medelci, I.E. Berishev, A. Bensaoula,* University of Houston

There is a great need for miniaturized, high power density, low cost capacitors that operate at high frequency and can sustain high operating temperatures. In our laboratory we are investigating the use of boron nitride (BN) based materials for such devices. Advantages of BN include high temperature and chemical resistance which should result in more compact, and reliable devices. We investigate here, the temperature stability of different multilayer capacitor heterostructures (e.g. TiN/BN/TiN/Si, Cu/BN/Cu/quartz, Cu/AlN/Si). To study the interdiffusion between the dielectric and the electrodes at elevated temperatures, we performed secondary ions mass spectroscopy (SIMS) on two structures: (I) TiN/B/BN/Si and (II) B/TiN/Si. In this manner we minimize ion mixing effects in determining the quality of the relevant interfaces (TiN/B/BN and B/TiN/Si) at annealing temperatures up to 500 °C. High resolution SEM pictures from TiN/BN/TiN/Si samples showed dense boron nitride layers with sharp interfaces. However Cu/AlN/Si structures showed copper diffusion into the silicon substrate due to the columnar structure of the single crystal AlN films. I-V measurements revealed the highly insulating properties of TiN/BN/TiN/Si capacitor. In fact a breakdown voltage (BDV) of 400 V/mm was measured for a 2000 Å BN thin layer. C-V measurements from a 3mm x 4 mm planar structure, show a capacitance value of 1.1 nF at 10 KHz and 1 Vrms. Furthermore the temperature dependence of C-V characteristics, dissipation factor, insulation resistance, and reliability of the ceramic capacitor will be presented. This work was supported by funds from a NASA cooperative agreement #NCC8-127 to SVEC, a Texas Advanced Technology Program Grant # 1-1-32061, and the Ballistic Missile Defense Organization/Science and technology and managed by William Shoup from The DTRA/CPTI. @FootnoteText@ *A. Bensaoula email address: Bens@jetson.uh.edu.

11:20am **EM-FrM10 The Deposition of Polycrystalline Si and SiGe by Ultra-high Vacuum Chemical Vapor Deposition System***, **K.M. Chen**, H.J. Huang,* National Chiao Tung University, Taiwan, R.O.C.*; L.P. Chen, G.W. Huang,* National Nano Device Laboratory, Taiwan, R.O.C.*; C.Y. Chang,* National Chiao Tung University, Taiwan, R.O.C.

The polycrystalline Si@sub 1-x@Ge@sub x@ (poly-Si@sub 1-x@Ge@sub x@) films have better properties than poly-Si for device fabrications, such as lower transition temperature and process thermal budget. For these reasons, the poly-Si@sub 1-x@Ge@sub x@ films have been utilized for low-temperature TFT fabrications and gate electrodes of MOS transistors. In this work, disilane and germane were used to grow poly-Si@sub 1-x@Ge@sub x@ films at low temperature (0.1). This implies that the growth mechanism of poly-Si@sub 1-x@Ge@sub x@ films is different from that of epitaxial Si@sub 1-x@Ge@sub x@ on Si. The uniformity of poly-Si@sub 1-x@Ge@sub x@ films depends on the Ge content, and it is improved by the addition of germanium. The result can be explained by the lower activation energy (< 0.25 eV) related to deposition of poly-Si@sub 1-x@Ge@sub x@, as compared to that of poly-Si (~2.1eV) in the surface-reaction limited growth mode. From the XRD and AFM analyses, the crystallinity and roughness of films are suitable to device fabrications.

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