Thin Films
Room 310 - Session TF+EM-WeM

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

Moderator: V.M. Bermudez, Naval Research Laboratory

8:20am **TF+EM-WeM1 In-situ Infrared Absorption Spectroscopy of High-k Dielectrics Growth on Semiconductors**, ***Y.J. Chabal***, *S. Rivillon, Y. Wang, K. Bratland, M.-T. Ho,* Rutgers University **INVITED**

Atomic Layer Deposition (ALD) is a particularly attractive method to grow a variety of heterostructures on semiconductors. It makes it possible for instance to deposit ultra-thin and near stoichiometric high-k dielectric metal oxides films one layer at a time in a highly conformal manner. For microelectronics applications, controlling the nature of the semiconductor/oxide interface and the oxide film itself is critical. For instance, formation of an interfacial SiO2 layer during metal oxide growth or incorporation of excess oxygen in the oxide is a major detriment to the performance of future MOSFETs. This talk discusses the use of in-situ infrared (IR) absorption spectroscopy to optimize silicon and germanium wafer pretreatments, to monitor interface formation during growth and to control the nature of metal oxides. For this work, a simple ALD reactor has been designed to be compatible with efficient transmission IR spectroscopy. The effects of surface chemical functionalization of H-terminated Si and Ge surfaces with Cl, NH3 prior to and thermal annealing after Al2O3 and HfO2 deposition have been investigated with emphasis on identifying the presence and bonding of oxygen at the interface. The mechanism for oxygen incorporation have been identified resulting in a much better control of the interface.

9:00am **TF+EM-WeM3 Real-time Sensing for Process Dynamics and Metrology in Tungsten Atomic Layer Deposition**, ***L. Henn-Lecordier***, *W. Lei, G.W. Rubloff,* University of Maryland

Atomic layer deposition (ALD) has been investigated using a novel wafer-scale reactor which features an internal mini-reactor and in-situ mass spectrometry (MS) for chemical analysis during tungsten ALD using WF6 and SiH4. Downstream MS sampling system measures deposition kinetics directly and reaction product MS signal is used for real-time thickness metrology. MS signal reveals ALD reactant and product species in real time through ALD process cycle, with product generation and reactant depletion indicative of species consumption. Both the H2 product from SiH4 exposure and the SiF4 product from WF6 exposure show the kinetics of self-limiting adsorption/reaction on the surface, which enables process optimization to minimize cycle time. MS data also directly indicates the influence of process temperature and precursor dose on film growth, and can also been employed for process optimization. The integrated reaction product MS signal over each exposure, when plotted against ALD cycle number, reveals different stages of ALD film growth and provides a quantitative measure of film thickness. Our study demonstrated that integrated reaction product MS signal has a good linear relationship with ALD film thickness. This provides a promising approach to advanced process control in ALD manufacturing.

9:20am **TF+EM-WeM4 Twin Boundaries can be Moved by Step Edges during Film Growth**, ***N.C. Bartelt***, *W.L. Ling, K.F. McCarty,* Sandia National Laboratories*; C.B. Carter,* University of Minnesota

Considerable effort has been devoted to minimizing twins in a film's microstructure because they typically degrade a film's performance. Twins are generally believed to originate from the nucleation stage of film growth. That is, when film islands nucleate, not all of them contain the same stacking sequence of film layers. Twin boundaries then occur where islands with different stacking sequence impinge. Attempts to reduce the density of twins are usually based on minimizing their nucleation or by removing them by annealing. We track individual twin boundaries in Ag films on Ru(0001) using low-energy electron microscopy (LEEM). We find that twin boundaries can move readily during film growth but relatively little during annealing. The growth-driven motion of twin boundaries occurs as film steps advance across the surface -- as a new atomic Ag layer approaches an fcc twin boundary, the advancing step edge carries along the boundary, which intersects the film thickness. This process can produce twin-free regions that are over 10 Âµm wide. These observations show that there can be a close connection between morphological evolution and microstructural evolution in thin films. This work was supported by the Office of Basic Energy Sciences, Division of Materials Sciences of the U.S. DOE under Contract No. DE-AC04-94AL85000.

9:40am **TF+EM-WeM5 In-Situ Real Time Spectroscopic Ellipsometry Studies of the Growth of Amorphous and Epitaxial Silicon for Photovoltaic Applications**, ***D.H. Levi***, *C.W. Teplin, E. Iwaniczko, Y. Yan, T.H. Wang, H.M. Branz,* National Renewable Energy Laboratory **INVITED**

In-situ monitoring of material properties during thin film deposition provides researchers with a valuable tool for maximizing solar cell performance, while also enabling efficient exploration of deposition parameter space. In this presentation I will describe how our research team at NREL has utilized in-situ real time spectroscopic ellipsometry (RTSE) to maximize our productivity in two related projects. We are using hot wire chemical vapor deposition (HWCVD) for low-temperature (90 to 350@super o@C) deposition of very thin films of amorphous hydrogenated silicon (a-Si:H) for a-Si / crystal-Si (c-Si) heterojunction (SHJ) solar cells. We are also using HWCVD for low temperature (200 to 440@super o@C) deposition of epitaxial films of silicon (epi-Si) on c-Si substrates. We utilize RTSE as both an in-situ diagnostic and a post-growth analysis tool for SHJ solar cells and epi-Si films grown by HWCVD. RTSE enables precise thickness control of the 3 to 10 nm thick layers used in the SHJ devices, as well as monitoring crystallinity and surface roughness in real time. Post-growth analysis of the RTSE data has enabled us to determine the optical, electronic, and structural properties of the thin films used in the SHJ devices, as well as crystallinity vs. thickness in the epi-Si layers. This information has been used to fine-tune the deposition parameters to optimize device performance and epi-Si thickness. Using input from RTSE analysis we have achieved a photovoltaic energy conversion efficiency of 17% on an Al-backed p-type float-zone c-Si wafer. Epi-Si films have been grown as thick as 500 nm utilizing parameter optimization based on RTSE analysis.

10:20am **TF+EM-WeM7 Analytic First-Order Solution for the Simultaneous Determination of Complex Refractive Indices and Thicknesses of Thin Films Deposited on Substrates**, ***I.K. Kim***, *D.E. Aspnes,* North Carolina State University

The problem of the simultaneous determination of the complex refractive index = n + i@kappa@ and thickness d of an isotropic thin film deposited on an isotropic substrate from polarimetric data (the so-called nkt problem) has never been solved analytically for a general , although solutions are available for the case of a transparent film (@kappa@ = 0). Here, we present an analytic solution for arbitrary n and @kappa@ that is valid to first order in d/@lambda@, where @lambda@ is the wavelength of light. The solution requires a knowledge of the change of pseudodielectric function <@epsilon@>, or alternatively the relative change @Delta@@rho@/@rho@ in the complex reflectance ratio @rho@, and either the relative change @Delta@Rp/Rp or @Delta@Rs/Rs, where Rp and Rs are the reflectances for p- or s-polarized light. Ranges of validity are obtained by comparing results to those obtained numerically using the exact three-phase-model expressions, and a procedure is presented for improving the accuracy. Numerical evaluation of the exact equations, which proceeds by least-squares analysis, is facilitated by using the first-order solution as a starting point. Depending on the sample the first-order solution is more effective at some energies than others, emphasizing the need for multiwavelength measurements over finite energy ranges. The most effective ranges can be determined from the correlation coefficients obtained in the least-squares analysis. As previously noted, the highest correlation generally occurs between n and d, although under some situations a high correlation can also occur between @kappa@ and d. The results should be particularly useful for systems involving reversible configurations, such as the cyclic adsorption and desorption of gases on clean surfaces, where accuracy can be improved by modulation techniques.

10:40am **TF+EM-WeM8 Linear Nanomechanical Measurements using a Novel AFM Technique**, ***P.M. Hoffmann***, Wayne State University **INVITED**

Atomic Force Microscopy has been extensively used to study roughness, adhesion and mechanical properties of surfaces and thin films. However, commonly used AFM techniques suffer from a variety of problems inherent to the technique: Snap-in instabilities in static AFM and non-linearities in dynamic AFM measurements. Here we present a new AFM technique that avoids these problems and is capable of stable, linear measurements of many systems, including atomic scale contact mechanics and atomic friction on surfaces. I will introduce the technique and present recent results, especially the finding that continuum mechanics applies in atomic contacts even if the number of atoms in the contact becomes very low and the observation of friction due to a single atomic defect.

Author Index

Bold page numbers indicate presenter