Thursday Morning, October 5, 2000

Thin Films
Room 203 - Session TF-ThM

Optical Films

Moderator: W.T. (Pat) Beauchamp, OCLI/JDSU

8:20am **TF-ThM1 Optical Thin Film Multilayer Systems for Thermal Emittance Modulation in the 300K Blackbody Spectral Region***, C.L. Trimble, E.B. Franke,* University of Nebraska, Lincoln*; J.S. Hale,* J.A. Woollam Company, Lincoln*; M. Schubert, **J.A. Woollam**,* University of Nebraska, Lincoln

We report on all-solid state electrochromic multilayer systems for thermal emittance modulation in the 300 K blackbody spectral region (2 to 40 microns). Amorphous and polycrystalline WO@sub 3@ thin films were used as ion storage and electrochromic layers, respectively. Tantalum oxide thin films were used as ion-conductor layers. All films were grown by magnetron sputtering. The multilayer system was switched between low and high emittance states by application of small voltages between two electrodes enclosing the stack, thereby moving previously electrochemically inserted Li@super +@ from the electrochromic to the storage layer and back. Two electrode designs were tested. One was built with an aluminum bottom layer electrode and an aluminum grid top electrode, and a second was made with aluminum grid electrodes on top and bottom. The optical constants of Li@super +@ intercalated and deintercalated tantalum oxide and WO@sub 3@ thin films were measured by ellipsometry from 2 to 35 microns. Prior to experimental layer stack formation, the thin film layer structure was optimized by calculations of emittance modulation based on the single layer optical constants and thicknesses. Performance of the layer stack was obtained by reflectance modulation @DELTA@R from 2 to 40 microns, and related to spectral emittance. Reflectance spectra were further used to calculate the emissivity modulation @DELTA@@epsilon@ integrated over a 300 K blackbody spectrum. Calculations also suggest application of thermal emittance modulating multilayers for temperatures up to 900 K. Additional simulations were performed assuming the layer stack covered by either a ZnS or a MgF@sub 2@ layer. Cover layers should protect the WO@sub 3@ layer, prevent Li@super +@ chemical reactions and moisture incorporation, and act as optical impedance match to improve switching performance. Supported by BMDO # DSAG60-98-C-0054, NASA Glenn Research Center grant # NAG3-2219, and NASA Epscor grant # NCC5-169.

8:40am **TF-ThM2 Structural Characterization of High and Low Index PECVD Optical Coating Materials: The Case of Titanium Dioxide and Silicon Dioxide***, **V. Hajek**, M. Latreche, J.E. Klemberg-Sapieha, L. Martinu,* Ecole Polytechnique, Canada*; A. Argoitia, W.T. Beauchamp,* Optical Coating Laboratory Inc.

In the present work we systematically study the optical, mechanical and structural characteristics of high index TiO@sub2@ and low index SiO@sub2@ films prepared by plasma enhanced chemical vapor deposition (PECVD) under controlled ion bombardment conditions. The films were obtained either individually from titanium tetrachloride (TiCl@sub4@), hexamethyldisiloxane (HMDSO), silicon tetrachloride (SiCl@sub4@) or from TiCl@sub4@/SiCl@sub4@ or TiCl@sub4@/HMDSO mixtures. Depositions were performed using radiofrequency (13.56 MHz) or dual-mode microwave/radiofrequency low pressure plasma systems; the latter one allows for a selective control of the ion energy and ion flux. In the first series of experiments we show that depending on the substrate bias voltage and temperature the refractive index for TiO@sub2@ at 550 nm varies from 2.20 to 2.40, the extinction coefficient remains low (k ~ 1-5 x 10@super-5@), and the film microhardness of about 10 GPa and low stress (0 - 200 MPa in compression) point to good film mechanical integrity. In the second series of experiments we study the transition from amorphous to crystalline structure, chemical bonding, and the role of chlorine in TiO@sub2@, SiO@sub2@ and mixed TiO@sub2@/SiO@sub2@ systems using ERD, XRD, FTIR, microraman, SEM and TEM techniques. The evolution of the microstructure is related to the energetic conditions during the film growth.

9:00am **TF-ThM3 Porous Thin Film Microstructure Control for Tuned Optical Properties***, **J.C. Sit**, S.R. Kennedy, M.J. Brett,* University of Alberta, Canada

The technique of glancing angle deposition (GLAD) has been used to produce highly porous thin film media by exploiting the enhanced atomic self-shadowing encountered with highly oblique angle deposition. Using advanced substrate motion with this technique allows for a great deal of control over the columnar morphology in these porous films. GLAD films with "helical" or chiral morphology have been shown to possess unique optical response including optical rotation and circular dichroism. As the structural parameters of the film determine many of the optical properties, careful control over the structural parameters is critical. For example, the wavelength at which peak optical activity is observed may be engineered to fall within the UV, visible, or NIR regions by appropriate control of the helical pitch. We present here characterization of the optical response with respect to several structural parameters in optical GLAD films including helical pitch and radius. Further, we apply similar characterization to several other microstructural types of interest such as periodically bent nematic (planar "s"-shaped) morphologies.

9:20am **TF-ThM4 Development of a PECVD Antireflective Layer for 193nm Polysilicon Gate Applications***, **W. Fan**, Y. Wang, K. MacWilliams, J. Tian, Z. Karim,* Novellus Systems, Inc., U.S.*; K. Ring,* Conexant Systems, U.S.

A PECVD silicon oxynitride film used as an antireflective layer (ARL) has been developed for patterning sub-0.15µm polysilicon gate at 193nm wavelength. PROLITH/2@super TM@ simulations were carried out to determine the range of optical parameters which results in minimum substrate reflectivity (e.g., a few tenths of one percent). An oxynitride film with n ~ 1.80, k ~ 0.60, and t ~ 260Å, which offers less than 0.1% reflectivity, can be successfully deposited by a typical sequential PECVD chamber. In conjunction with achieving a film with these parameters, film deposition process window, film measurement variation, and post-treatment effects were taken into consideration to ensure minimal reflectivity. Finally, the films were patterned with 193nm photoresists. Cross-section SEM shows tight CD control.

9:40am **TF-ThM5 Characterizing Bi-Layer ARC for Advanced Lithography***, **I. Bloomer**, D.V. Likhachev, J. Lam, D. Harrison,* n&k Technology

Bi-layer ARCs consisting of Si@sub3@N@sub4@/Si-rich SiN@subx@/Si-substrate are gaining attention for advanced lithography applications. Such bi-layer ARCs are more effective than single-layer ARCs in reducing standing waves, and furthermore provide wider processing latitude for overall ARC thickness. The processing conditions for a stoichiometric film such as Si@sub3@N@sub4@ are well controlled, and therefore its index of refraction, n, and the extinction coefficient, k, are well established. By contrast, the n and k values of a silicon-rich SiN@subx@ film are variable and strongly dependent on the gas distribution of the processing chamber. Typically the output is a film with non-uniform composition along the z-axis. For such bi-layer ARCs, rapid and accurate characterization is imperative to produce a consistent, uniform product. We will present a metrology technique, based on the 'n&k Method', that accurately and rapidly characterizes bi-layer ARCs despite variations and non-uniformities. With this method, the thicknesses of both the Si@sub3@N@sub4@ and the non-uniform silicon-rich SiN@subx@ layers are determined simultaneously, along with the n and k spectra (190 to 1000nm) of the non-uniform silicon-rich SiN@subx@ layer, which is determined as a function of z. The â€œn&k Methodâ€ is based on broad-band reflectance that incorporates all-reflective optics to provide a signal-to-noise ratio of better than 0.2% over the entire measured wavelength range of 190 to 1000nm. The Forouhi-Bloomer model for n and k is used to analyze the raw data. Results for a series of samples with silicon-rich SiN@subx@ thicknesses ranging from 180 to 300 Å and the Si@sub3@N@sub4@ thicknesses ranging from 1000 to 1300 Å will be presented, along with the results of the z-direction compositional non-uniformity of the silicon-rich SiN@subx@ layer. We will also discuss how these results can be used to optimize the manufacturing process for bi-layer ARCs.

10:00am **TF-ThM6 A Near-grazing-incidence, Antireflective Coating for 121.6 nm for a Mars-bound Neutral Particle Detector.***, **S. Olson**, D.D. Allred, M.B. Squires, D. Markos, C.E. Mills, R.T. Turley,* Brigham Young University

A neutral particle detector is being planned to orbit Mars as part of the ASPERA mission. It is designed to detect and classify atoms in the exosphere of Mars by their identity and velocity using a time-of-flight mass spectrometer. An electron released on collision with the first surface in the spectrometer (the start surface) initiates a start pulse for the spectrometer. The most significant source of noise is thought to be UV photons-particularly hydrogen Lyman alpha (121.6 nm), which are capable of generating false start and stop pulses. We have design using a genetic algorithm and fabricated a coating for the START surface which absorbs UV photons at 121.6 nm thus preventing their reaching the stop surface. The particular optical challenge was the fact that the particles and incident light arrives at an angle of 10 to 20 degrees from grazing. We will discuss the use of a genetic algorithm in designing the coating, its fabrication, AFM measurements of sample and at-wavelength measurements of their reflectivity.

10:20am **TF-ThM7 Studies of Pulsed DC Power in Magnetron Sputtering Systems*, P.J. Kelly,*** *J. O'Brien,* University of Salford, UK*; J.W. Bradley,* UMIST, UK, U.K.*; R. Hall, G. Roche,* Advanced Energy Industries Inc.*; R.D. Arnell,* University of Salford, UK

The application of pulsed DC power has proved to be highly advantageous in magnetron sputtering processes. Indeed, reactive pulsed magnetron sputtering is now amongst the leading techniques for the deposition of dielectric films. When the magnetron discharge is pulsed in the mid-frequency range (20-350kHz), the periodic voltage reversals effectively discharge poisoned regions on the target. This significantly reduces the occurrence of arc events at the target and stabilises the deposition process. As a consequence, substantial improvements have been observed in film structure and properties, compared to films produced by continuous DC processing. More recently, interest has also focused on the effects which can arise when pulsed DC power is applied to the substrate. Pulsing the bias voltage appears to significantly increase the substrate ion current. For example, when compared to DC biasing, preliminary results have shown that a three-fold increase can be achieved in the current drawn at the substrate if the bias voltage is pulsed at 350kHz. However, despite these successes, aspects of this technology are not yet well understood. Consequently, detailed studies of the application of pulsed DC power, both to the magnetron and to the substrate, in a dual, variable field strength closed-field unbalanced magnetron sputtering system, are now in progress. Initial findings are reported here.

10:40am **TF-ThM8 Art and Science of Fabricating Optical Coatings by PECVD*, L. Martinu,*** Ecole Polytechnique, Canada*; D. Poitras,* National Research Council of Canada, Canada **INVITED**

Low pressure plasma deposition using gas phase precursors is being increasingly used for the fabrication of dielectric optical films and coatings. This includes single-layer, multilayer, graded index and nanocomposite optical thin film systems for applications such as optical filters, antireflective coatings, optical waveguides and others. Besides their optical properties (refractive index, extinction coefficient, optical loss), these systems very frequently offer other desirable "functional" characteristics such as hardness, scratch-, abrasion- and wear resistance, adhesion to various technologically important substrate materials such as polymers, hydrophobicity or hydrophilicity, long-term chemical, thermal and environmental stability, and others. In this presentation, we review advances in the development of plasma processes and plasma systems, in the synthesis of high- and low index optical thin film materials, and in the control of plasma-surface interactions leading to desired film microstructures. We particularly underline the specific aspects of PECVD which are different from other conventional techniques used for optical films; this includes in particular fabrication of graded index (inhomogeneous) layers, high deposition rate, control of interfaces, enhanced mechanical and other functional characteristics, and industrial scale-up.

11:20am **TF-ThM10 Inhomogeneous Optical Coatings Deposited by Plasma*, D. Poitras,*** National Research Council, Canada*; L. Martinu,* Ecole Polytechnique, Canada

Inhomogeneous optical coatings with a refractive index continuously varying with thickness appear to offer several attractive features from both fundamental and application points of view: (1) Understanding how accidentally-generated inhomogeneities can affect the optical performance of coatings can help to optimize the performance of optical filters; (2) purposely introduced and controlled optical inhomogeneities can be used to fabricate high performance inhomogeneous optical systems such as rugate filters; and (3) coatings with graded interfaces generally exhibit higher mechanical strength, better adhesion and good long term stability. In the present work, we studied these different aspects of inhomogeneous coatings produced by plasma enhanced chemical vapor deposition (PECVD). We used admittance diagrams (i) to interpret recent results concerning the effect of transition layers on the reflectance spectra of homogeneous single films, and (ii) to derive an alternative explanation of specific properties of rugate filters. In addition, we describe a simple way for introducing the refractive index dispersion while designing rugate filters. The latter approach is documented by the performance analysis of complex refractive index profiles in a double band rugate filter and in a chirp mirror fabricated by PECVD.

11:40am **TF-ThM11 Uranium Coated Optics for Space Applications in the Extreme Ultraviolet*, D.D. Allred,*** *R.T. Turley,* Brigham Young University*; W.C. Cash,* University of Colorado*; M.B. Squires, D. Oliphant,* Brigham Young University

We have developed a new family EUV multilayer mirror coatings using uranium. Using this approach we have coated a set of 6 mirrors for the EUV Imager, a component of the IMAGE mission. It will study the distribution of He + in the Earth's plasmasphere by detecting its resonantly scattered emission at 30.4 nm (41 eV). The 30.4 nm feature is, in principle, relatively easy to measure because it is the brightest ion emission in the plasmasphere, it is spectrally isolated and the background at that wavelength is negligible. There is, however, a bright emission at 58.4 nm (21 eV) light, which comes from neutral helium in the earth's ionosphere which also must be blocked. It is at too high an energy to filter with aluminum but at too low an energy to have negligible reflectance from most materials commonly used in EUV mirrors. Thus, a multilayer system which satisfied two optical functions: high reflectance (>20%) at 41 eV but low reflectance (

Author Index

Bold page numbers indicate presenter