Wednesday Morning, November 5, 2003

Thin Films
Room 329 - Session TF-WeM
Optical Thin Films and Photovoltaics I
Moderator: R. Sargent, OCLI

8:20am **TF-WeM1 Multilayer Optical Coatings Using Closed Field Magnetron Sputtering***, **J.M. Walls**, D.G. Gibson, J. Hampshire, D.G. Teer,* Applied Multilayers Ltd, UK

Magnetron Sputtering has many advantages for the deposition of multilayer optical coatings. The process operates at high energy producing dense, spectrally stable coatings. The sputtering process is also "cold", making it suitable for use on the widest range of substrates including polymers. This paper will describe a new process that allows high quality, multilayer metal-oxide thin films to be deposited at high rates using Closed Field magnetron sputtering together with pulsed dc power. The Closed Field process for optical coatings uses two or more different metal targets. The target is held in a partially oxidised state controlled using plasma emission monitoring and the oxidation occurs in the entire volume around the rotating substrate carrier. In addition to describing the new process, this paper will discuss the optical properties of individual layers and their application to a range of multilayer precision optical coatings. The ion current density and the low bias voltage provided by Closed Field magnetron sputtering produces films at a high rate with excellent optical properties. Machines based on the Closed Field are scaleable to meet a range of batch size requirements. Examples of multilayer coatings in the visible and infra-red spectra will be provided. Examples in the visible spectrum will include Anti-reflective and other coatings using fully oxidised SiO2, TiO2 ITO and other metal-oxide films. Applications in the infra-red will incorporate materials such as Si, Si3N4 and TiO2. Typically, thin film thickness control is accomplished simply using time although quartz crystal monitoring or optical monitoring are used for more demanding applications. Fine layer thickness control is also assisted with a specially designed rotating shutter mechanism.

8:40am **TF-WeM2 Smooth Optical Thin Film Formation by Oxygen Cluster Ion Beam Assisted Depositions***, **N. Toyoda**, I. Yamada,* Himeji Institute of Technology, Japan

Ta@sub 2@O@sub 5@/SiO@sub 2@ and Nb@sub 2@O@sub 5@/SiO@sub 2@ were deposited with oxygen gas cluster ion assisted deposition at low-temperature for optical filters. As one cluster ion has thousands of O@sub 2@ molecules, equivalently low-energy ion irradiations are realized at several keV of total acceleration energy. Due to the dense energy deposition of cluster ions, high-temperature and high-pressure conditions are realized at the impacted area, which enables to deposit high quality thin films without heating the substrate. Also, GCIB shows significant surface smoothing effects, which realizes very flat surface and interfaces for multi-layered structures. In this study, O@sub 2@-GCIB was applied to form high quality optical films. With gas cluster ion assisted deposition, high refractive index and very uniform amorphous structures were observed with cross-sectional SEM. The surface or interfaces of Ta@sub 2@O@sub 5@/SiO@sub 2@ films were also very flat by surface smoothing effect of cluster ion beams. Even though the deposited surface was rough (average roughness 1.5nm), the surface roughness of the deposited Ta@sub 2@O@sub 5@ film was improved to 0.7nm. As there is strong surface smoothing effect with O@sub 2@ cluster ion beam assisted deposition at low substrate temperature, it is appropriate to form multi-layered optical filters.

9:00am **TF-WeM3 Scandium and Vanadium Multilayer Mirrors: Working Towards High Reflectivity in the Extreme Ultraviolet***, G. Acosta, **D. Allred**, R. Davis,* Brigham Young University

Despite bulk reflectivities of materials in the EUV being typically less than 7%, it is possible to design a multilayer mirror using thin films to achieve reflectivities in the vicinity of 30-40%. Inspired by the 1998 Uspenski paper@footnote 1@ which theorized 72% reflectance of 42 nm light, we have been working on developing a design scheme that uses the rare earth metal scandium to achieve such high reflectivies. For the multilayer coating, we chose to pair scandium with vanadium to ensure distinct interfaces between the materials, since scandium and vanadium are immiscible. Our thin film samples (typically 1.5-10 nm thick) were characterized with Atomic Force Microscopy, Ellipsometry, and using an Extreme Ultraviolet Scanning Monochrometer for reflectivity measurements. In addition to preliminary EUV reflectivity predictions, optical constants were found experimentally over the 800-400nm range, as well as in the EUV. @FootnoteText@ @footnote 1@ Uspenski et al, Optics Letters, vol. 23, no. 10, 771.

9:20am **TF-WeM4 Improvement of Reproducibility in Deposition Rate of MgF@sub 2@ Film Prepared by an rf Sputtering Technique named Keep Molecular Sputtering Method***, K. Kawamata, **T. Deguchi**,* Olympus Optical Co., Ltd., Japan*; E. Kusano, A. Kinbara,* Kanazawa Institute of Technology, Japan

MgF@sub 2@ film is generally formed by electron-beam evaporation rather than sputtering, because sputtered MgF@sub 2@ film shows poor transparency, resulting from F deficiency. To solve this problem, we have proposed a Keep-Molecular-Sputtering (K-M-S) method. It involves keeping magnesium fluoride target at a high temperature, and providing the sputtering species as a form of molecules. However, deposition rate of the K-M-S method has a large dispersion. In this study, reproducibility of deposition rate of MgF@sub 2@ film in the K-M-S method has been improved by monitoring optical emission intensity ratio of MgF@super *@/O@super *@. A sputtering-up-type rf magnetron sputtering machine was used in the experiment. Sputter source is 1-2 mm granular MgF@sub 2@ put on a 100 mm diam. quartz plate backed with a Cu plate. The source-to-substrate distance was 75 mm. Discharge gas was O@sub 2@ with a flow rate of 80 sccm. MgF@sub 2@ films are deposited on glass substrate (BSL7) set to an aluminum holder rotating during film deposition. Pre-sputtering of the target started when H@sub 2@O partial pressure became constant. Rf power was controlled to a certain value in the range between 480 and 520 W to remain the optical emission intensity ratio of MgF@super *@/O@super *@ at a constant value. The reproducibility of the deposition rates has been improved to ±17%(115±24 nm/min.) by controlling rf power, compared with ±28%(111±31 nm/min.) obtained for a constant power of 550W. For all MgF@sub 2@ films, optical absorptance at a wavelength of 400 nm was less than 5%. We have also discussed mechanisms of the K-M-S process based on analysis using a quadrupole mass spectrometer.

9:40am **TF-WeM5 Optical Properties and Microstructure of Plasma Deposited Ta@sub 2@O@sub 5@ and Nb@sub 2@O@sub 5@ Optical Thin Films***, H. Szymanowski, J.-P. Masse, **O. Zabeida**, J.E. Klemberg-Sapieha, L. Martinu,* Ecole Polytechnique of Montreal, Canada

Advanced optical filter applications require not only an appropriate control of the optical constants of the thin films but also a suitable control of other film properties such mechanical performance, thermal and environmental stability, absence of refractive index inhomogeneities and others. In this respect, plasma enhanced chemical vapor deposition (PECVD) allows one to fabricate films with low as well as with high refractive index, and it also offers a possibility for stress compensation, control of refractive index gradients, and high deposition rates at low substrate temperature. In the present work we study the characteristics of two high index optical materials, namely amorphous tantalum pentoxide (Ta@sub 2@O@sub 5@) and niobium pentoxide (Nb@sub 2@O@sub 5@) obtained by PECVD, respectively, from penta-ethoxy tantalum Ta@sub 2@(OC@sub 2@H@sub 5@)@sub 5@ and penta-ethoxy niobium, Nb@sub 2@(OC@sub 2@H@sub 5@)@sub 5@, precursors. We particularly investigated the effect of the energetic conditions on the film growth by using different modes of plasma excitation, namely radio frequency (RF), microwave (MW) and dual-mode microwave/radio frequency (MW/RF) discharges. Under sufficient ion bombardment, controlled by the RF-induced negative substrate bias, the dense Ta@sub 2@O@sub 5@ and Nb@sub 2@O@sub 5@ films exhibited a refractive index of 2.16 and 2.26 (at 500 nm), respectively, while the extinction coefficient was below 10@super -5@, as determined by spectroscopic ellipsometry, spectrophotometry and thermal deflection optical calorimetry. We found that increasing ion bombardment during the film growth leads to an appreciable increase of carbon concentration incorporated in the films, as indicated by a strong double peak at 1400 and 1500 cm-1 in the FTIR spectra. Elastic recoil detection (ERD) measurements reveal an atomic concentration of 2.5% and 5,5% of carbon in the bulk of the Ta@sub 2@O@sub 5@ and Nb@sub 2@O@sub 5@ films. The presence of carbon did not appear to negatively affect the film optical and mechanical performance and stability. We discuss the possible mechanism of carbon bonding in these films in a form of metal chelate and bridging groups.

10:00am **TF-WeM6 The Anneal Behavior of Reactively Sputtered HfN Films**, *J. Lannon Jr., C.C. Pace, S. Goodwin,* MCNC Research and Development Institute; *S. Solomon,* Acumen Consulting; *P. Bryant, J. Oleson,* Santa Barbara Infrared, Inc.

This article reports electrical, optical, structural and thermo-physical properties of reactively sputtered HfN films with respect to elevated-temperature annealing. All metal nitride films were sandwiched between sputtered Si3N4 films. The resulting reduction in electrical resistivity with anneal is explained by a combination of XPS, RBS and SIMS analyses, and the physical mechanisms responsible for the observed anneal behavior are discussed. The negative TCR is also explained. Infrared optical properties of these film stacks were investigated and found to show the expected correlation with electrical properties, while the anneal behavior was found to exhibit anomalies that were independent of the as-deposited properties.

Author Index

Bold page numbers indicate presenter