## Thin Films Division
## Room 310 - Session TF-MoM

### Transparent Conductive Oxides
**Moderator:** T.J. Coutts, National Renewable Energy Laboratory

8:20am **TF-MoM1 Atmospheric Pressure Chemical Vapor Deposition of Transparent Conducting Films of Fluorine-Doped Zinc Oxide**, *H. Liang,* ***R.G. Gordon,*** Harvard University **INVITED**

Transparent conducting fluorine doped zinc oxide was deposited as thin films on soda lime glass substrates by atmospheric pressure chemical vapor deposition (CVD) at substrate temperatures of 480@super o@C to 500@super o@C. The precursors diethylzinc, tetramethylethylenediamine and benzoyl fluoride were dissolved in xylene, and this solution was nebulized ultrasonically and then flash vaporized by a carrier gas of nitrogen preheated to 150 @super o@C. Ethanol was vaporized separately, and these vapors were then mixed to form a homogeneous vapor mixture. Good reproducibility was achieved using this new CVD method. Uniform thicknesses were obtained by moving the heated glass substrates through the deposition zone. The best electrical and optical properties were obtained when the precursor solution was aged for more than a week before use. The films were polycrystalline and highly oriented with the c-axis perpendicular to the substrate. More than 90% of the incorported fluorine atoms were electrically active as n-type dopants. The electrical resistivity of the films was as low as 5x10@super -4@ ohm-cm. The mobility was about 45 cm@super 2@/V-s. The electron concentration was up to 3x10@super 20@/cm@super 3@. The optical absorption of the films was about 3-4% at a sheet resistance of 7 ohms/square. The diffuse transmittance was about 10% at a thickness of 650 nm. Amorphous silicon solar cells were deposited using the textured fluorine doped zinc oxide films as the front electrode. The short circuit current was increased over similar cells made with fluorine doped tin oxide.

9:00am **TF-MoM3 Effects of Layered Structure on Properties of Transparent Conductive Films of ZnO/ZnO:Al**, ***K. Tominaga,*** *T. Murayama, Y. Sato, I. Mori, T. Ushiro, T. Moriga, I. Nakabayashi,* The University of Tokushima, Japan

Recent data for transparent conductive films show that film resistivity seems to saturate at 10@super -4@ ohm-cm or above. This circumstance is common in ZnO and ITO films. To overcome this difficulty, approaches different from conventional parameter controls should be examined. In ZnO film preparation, we tried additional Zn adding during the film deposition. Recent investigations showed that the additional Zn during the sputtering of ZnO:Al target (doped 2 wt% aluminum oxide) induced an increase of both carrier concentration and Hall mobility. As the result, film resistivity was decreased. This suggested that an incorporation of oxygen deficient ZnO phase (ZnO:O@sub v@) in ZnO:Al phase improves the film crystallinity of ZnO:Al and decreases defects in ZnO:Al. This effect may be observed in general for the case of layered films of ZnO:Al and ZnO. To confirm this, we produced a multilayered film of ZnO:Al and ZnO:O@sub v@ film and investigated the role of the inserted ZnO:O@sub v@ layer in electrical conduction and optical property. Films were deposited by alternative sputtering method, where a ZnO:Al(2 wt%) target and a conductive ZnO target which only contains native donors were sputtered alternatively in pure Ar gas at 1 mTorr for a definite time. This process was succeeded to deposit a definite film thickness. The results showed that the film resistivity is decreased by inserting ZnO:O@sub v@ layer. This is due to an improvement of doping efficiency of Al donors in ZnO:Al layer by inserting ZnO:O@sub v@, in addition to carrier redistribution between two layers.

9:20am **TF-MoM4 Chemical State Effects on Doped ZnO Film Properties**, ***G.J. Exarhos,*** *L.-Q. Wang, C.F. Windisch, Jr.,* Pacific Northwest National Laboratory

Zinc oxide is representative of the class of transparent conducting oxides which exhibit high transmission at visible wavelengths and concurrent low electrical resistivity. The resident conductivity and associated long wavelength reflectivity of these II-VI semiconductor films arises from the introduction of defect levels within the bandgap generated during the deposition process itself or during subsequent processing. In this work, films are prepared by means of rf-sputter and solution deposition methods. The deposition parameters are varied in order to increase conductivity in films which incorporate multivalent cationic dopants (Ga@super +3@, In@super +2@, Cu@super +2@, Au@super +3@, Pt@super +4@,...) within the wurtzite lattice. Post deposition modification of films on silica, Si, Al, or Pt substrates involves annealing in Ar/4% H2 or cathodic reduction in an electrochemical cell. Electrochemical film modification is carried out in aqueous solution (pH 7) or in an organic solvent such as CH@sub 3@CN. As-deposited and modified films are characterized using a cadre of analytical methods including XPS, AFM, TEM, XRD, Raman spectroscopy, and Electrochemical Impedance Spectroscopy. EIS measurements enable selective characterization of polarization effects within the oxide film and localized chemistry at the film-solution interface as a function of applied potential. The defect structure of the oxide is readily probed by means of in situ Raman spectroscopy during electroreduction. Results indicate that the LO Raman E@sub 1@ mode intensity, linewidth, and resonance frequency are particularly sensitive to the nature and concentration of defect states present in the film. Based upon these studies, a surface hydroxyl species is proposed to explain the observed reversible changes in conductivity. Such measurements complement the XPS studies which probe dopant oxidation state. Insight into film properties stability is based upon the electrochemical studies and measured variations in film properties upon subsequent annealing.

9:40am **TF-MoM5 Photoemission Spectroscopy Analysis of ZnO Films for Display Applications**, *E.W. Forsythe, Y. Gao,* University of Rochester; ***G.S. Tompa,*** *L.G. Provost,* Structured Materials Industries, Inc.; *J. Doyle,* Advanced Display Systems

An important factor in the performance of most displays is the quality of at least one transparent conductive oxide contact layer. Presently, indium tin oxide (ITO) is predominantly used because it is an accepted standard and has a great deal of invested development. However, for several reasons, including work function, physical stability, and band alignment, ITO is not always the ideal contact layer for a given display technology. We will report the physical properties of ZnO based films prepared by metal organic chemical vapor deposition (MOCVD) using ultraviolet and x-ray photoemission spectroscopy (UPS and XPS). In addition, the surface characteristics of the films are modified with series of cleaning and polishing steps. Using UPS, the work function for ZnO is 4.23eV. XPS results show an oxide layer more than 5nm on the surface of the as-received MOCVD films, with a small fraction of Zn and Ga. This oxide layer is removed by a cleaning and plasma treatment, which enhances the conductivity of the ZnO films. Finally, we will report atomic force microscopy results before and after cleaning and polishing as well as chemical etching results. This work was supported in part by DARPA DAAL01-96-K-0086, NSF DMR-9612370.

10:00am **TF-MoM6 Transparent and Conductive Multicomponent Oxide Films Prepared by Magnetron Sputtering**, ***T. Minami,*** Kanazawa Institute of Technology, Japan **INVITED**

Recently, multicomponent oxides composed of combinations of binary compounds or ternary compounds have attracted much attention as new materials for transparent and conductive thin films. This paper introduces transparent conducting multicomponent oxide films prepared with varied chemical compositions by magnetron sputtering. It was found that most multicomponent oxides composed of combinations of binary compounds contained at the least one ternary compound; highly transparent and conductive films could be prepared in the ternary compound. In addition, if binary or ternary compounds which produced transparent and conductive films when prepared by magnetron sputtering were used as the starting materials, transparent and conductive films composed of combinations of these binary or ternary compounds could be also produced from all compositions in the resulting multicomponent oxides. In addition, it was found that most of the properties of transparent conducting multicomponent oxide films were mainly determined by the metal element contained in the oxide. It can be concluded that transparent conducting multicomponent oxide films are suitable for specialized applications because their electrical, optical and chemical properties as well as physical properties such as band-gap energy, refractive index and work function can be controlled by changing the chemical composition.

10:40am **TF-MoM8 Improvement of Microstructure of Indium-Tin-Oxide Films by Thin Film and Surface Technologies**, ***Y. Taga,*** *T. Satoh, M. Ishii, T. Ohwaki,* TOYOTA Central Research & Development Labs., Inc., Japan

A rapid progress has been made in the practical device applications of flat panel displays (FPD) such as liquid crystal display, electroluminescence display, etc. In accordance with this trend, strong demands have been actually appeared in the quality of transparent conductive films such as indium-tin-oxide (ITO). Up to now, a lot of studies have been devoted mainly to the electrical and optical properties of ITO films. However, it

becomes clear that microstructure of ITO films gives an important influence on the performance and durability of FPD's. In this paper, we tried to improve the microstructure of ITO films by controlling the sputter deposition and plasma treatment conditions in thin film preparation. Furthermore, we investigated the effect of microstructure of ITO films on the electrical properties of Ta-Sn-O films in the system of Al/Ta-Sn-O/ITO layered structure in inorganic electroluminescence displays. It was found that the microstructures of ITO films changed drastically with oxygen partial pressure in Ar-O@sub 2@ sputtering gas; i.e., with increasing oxygen partial pressure, grain size become small and surface morphology become smooth. Measurements of I-V characteristics in Al/Ta-Sn-O/ITO revealed that the leakage current at low electric field has been diminished by the improvement of microstructure of ITO films.

11:00am **TF-MoM9 Work Function Modification of Indium Tin Oxide**, ***S.M. Tadayyon***, *K. Griffiths, P.R. Norton,* University of Western Ontario, Canada*; C. Tripp, Z. Popovic,* Xerox Research Centre of Canada, Canada

Indium-tin-oxide (ITO) is a transparent conducting material widely used in electronic devices such as flat panel displays, solar cells, IR detectors and OLED's where it is used as the hole injecting electrode. It is desirable to have an electrode possessing as high a work function as possible, and so extensive investigations into properties and modification of ITO have been undertaken. The objective of the present study is the possible modification of ITO work function (WF) using evaporated metal overlayers. We have therefore studied gold overlayers because of the high WF of gold, and its inertness. ITO films on glass substrates (resistance ~ 10 Ohms/sq) were cleaned with a UV-ozone treatment before entry into a UHV system. Auger electron spectroscopy (AES) was used to determine the surface composition. The change of work function (WF) of the surface (± 10mV) was determined by a Kelvin probe technique and correlated with the absolute coverage (@theta@) of Au in the range 0<@theta@ 5x10@super 15@ Au cm@super -2@. The final WF values were not very different from the non-UV-ozone treated samples. Studies on bulk Au and ITO covered by Au in a single deposition, showed that the low final WF value in the sequential experiments was attributable to adsorption of hydrocarbons from the 5x10@super10@ torr vacuum, and that useful increases in WF are attainable on transparent ITO electrodes

11:20am **TF-MoM10 Application of High Speed Four-Parameter Stokes Vector Spectroscopy to the Characterization of Textured and Specular Transparent Conducting Oxide Thin Films**, ***P.I. Rovira***, *R.W. Collins,* Pennsylvania State University

A rotating-compensator multichannel ellipsometer has been used to measure the four unnormalized Stokes vector elements associated with the polarization state of polarized light reflected from both specular and textured transparent conducting SnO@sub 2@:F films. This technique provides not only the ellipsometry angles (@PSI@, @DELTA@), but also the reflectance R and the degree of polarization p. With a photodiode array detector, spectra in (@PSI@, @DELTA@), R, p) having 100 points from 1.5 to 3.75 eV can be collected with a minimum acquisition time of ~32 ms. In contrast to rotating-polarizer multichannel ellipsometry which tends to be inaccurate when @DELTA@ equals 0° or ±180°, or p<1, the rotating-compensator approach provides high accuracy measurements of the phase shift @DELTA@ over its full range (-180° to 180°) even when p<1. Therefore, this new configuration permits us to make accurate ellipsometric measurements for SnO@sub 2@:F films on glass substrates, which is the structure of choice for large area device applications such as photovoltaics. In addition to conventional microstructural characterization using the ellipsometry angles (@PSI@, @DELTA@), we have incorporated light scattering due to the textured surfaces into the analysis using the reflectance and degree of polarization. From the latter analysis, information on the SnO@sub 2@:F texture can be extracted. The results are consistent with direct images by scanning electron microscopy and atomic force microscopy. A comparison of the degree of polarization measured for the specular and textured SnO@sub 2@:F films suggests that deviations in p from unity for the latter are due to the detection of light multiply-scattered by the texture into the specular direction. Finally, the rotating-compensator multichannel ellipsometer developed here can be readily adapted to real time analysis of solar cells prepared on textured transparent conducting oxide films in commercial processes.

11:40am **TF-MoM11 Transparent Conductive Oxides with Improved Performance for Plastic Flat Panel Displays**, ***C.I. Bright***, Delta V Technologies

The major use of Transparent Conductive Oxides (TCO) is in Flat Panel Displays (FPD). One of the major issues preventing the use of a plastic film substrate for FPD is its moisture and oxygen permeability. The permeation of water and oxygen limits the long-term stability of the display device. Another issue with plastic substrates is the low conductivity of the TCO that must be deposited at a low temperature. The Polymer MultiLayer (PML) process for vacuum evaporation of organic monomers and in-situ e-beam or UV polymerization has been shown to produce excellent substrate smoothing and when combined with other layers, outstanding barrier properties on plastic films, e.g., polypropylene and polyester (PET). Oxygen and moister permeation rates for an aluminum film with PML base coat, are one to two orders of magnitude lower than with just an aluminum layer alone. Similarly, permeation rates with PML plus an aluminum oxide layer, are another one to two orders of magnitude lower than PML with aluminum. Thus, it is proposed to combine a PML base coat and the necessary TCO transparent electrode layer, to form a barrier and solve the permeability problem of plastic substrates. This PML base coat layer should also provide a pristine surface for nucleation of the deposited TCO. Therefore, the surface resistivity of the TCO should be lower, for a given film thickness, due to its improved microstructure. The experimental results for an evaporated acrylic base coat on PET substrate, follow by DC sputtering of ITO from a ceramic target in a single pass through a web coater, are reported. Results for ITO sputtered directly onto the PET substrate without the smoothing base coat are also reported. The optical, electrical and barrier properties for both constructions were measured and compared. Three-layer constructions are also considered with, for example, a silicon dioxide barrier layer deposited either onto the PET substrate, TCO or on the base coat. The potential benefits of these configurations are compared with the two-layer configuration results reported.

## Vacuum Metallurgy Division
## Room 328 - Session VM+TF-MoM

### Ionized-PVD: Processes, Properties, and Applications

**Moderator:** S.L. Rohde, University of Nebraska, Lincoln

8:20am **VM+TF-MoM1 Ionised PVD and Filtered Arc Deposition; Processes, Properties and Applications**, ***P.J. Martin***, *A. Bendavid,* CSIRO, Australia*; H. Takikawa,* Toyohashi University, Japan **INVITED**

Recent innovations in vacuum arc deposition have resulted in the development of the filtered arc source as a deposition tool for a range of technologically important materials. The vacuum arc was recognised early on as a potentially useful source of energetic, ionised material and a practical high rate method for depositing thin films with bulk properties and the deposition of new materials. The inherent problem of microdroplet contamination was overcome by several approaches, the toroidal magnetic duct being the most prevalent. The present state of the art of filtered arc deposition (FAD) is discussed in terms of the current understanding of the emitted fluxes, the properties of the materials deposited by these devices and new applications.

9:00am **VM+TF-MoM3 Transport of a Cathodic-Arc Plasma Through a Linear-Solenoid Macroparticle Filter**, ***B.P. Cluggish***, *B.P. Wood,* Los Alamos National Laboratory

A long standing problem in the use of cathodic arcs for deposition of coatings is the production of micron sized droplets, or "macroparticles," of the cathode material. These macroparticles hit and stick to the substrate, causing defects in the coating. One widely used method for "filtering out" the macroparticles is to guide the arc plasma through a solenoidal "magnetic duct" (a metal tube with an axial magnetic field.) The macroparticles travel in straight lines and thus hit and stick to the walls of the duct, rather than reaching the substrate. Unfortunately, most of the plasma ions are lost as well. For this reason, we are performing measurements to understand the transport of the plasma through a duct. The ion flux is found to decay exponentially along the length of the duct, and the magnetic field is crucial for reducing the ion losses. However, increasing the field strength above 50 G has no effect on the ion losses. Furthermore, unlike previous researchers @footnote 1@, we find that applying a positive voltage to the duct has little effect on the ion losses. We have developed a computer simulation which reproduces our results, and predicts that the injection conditions at the entrance to the duct are crucial in determining the ion losses. This work supported by the U.S. D.O.E. @FootnoteText@ @footnote 1@ A. Anders, S. Anders, and I. G. Brown, J. Appl. Phys., vol. 75, pp. 4900-4905, 1994

9:20am **VM+TF-MoM4 Characterization of Magnetron-Sputtered Partially Ionized Deposition as a Function of Metal and Gas Species**, ***M.M.C. Allain***, *D.B. Hayden, D.R. Juliano, D.N. Ruzic,* University of Illinois, Urbana-Champaign

A dc planar magnetron with a 33-cm diameter target is coupled with a secondary plasma source to ionize the sputtered metal neutral flux to control the angular distribution of the flux arriving at the surface of the substrate. The secondary radio-frequency (rf) plasma is created between the sputtering target and the substrate by a multi-turn coil located in the vacuum chamber. The rf plasma increases the electron density, which results in significant ionization of the neutral metal flux from the sputtering target. By applying a small negative bias to the substrate, metal ions are drawn to the substrate at normal incidence. A gridded energy analyzer and a quartz crystal microbalance (QCM) are attached to a pulley system that allows the ion and neutral deposition rates to be determined along the substrate plane. The ionization fraction of the flux incident onto the QCM can then be determined as a function of position. The ionization rate is a sensitive function of the metal's ionization potential (IP). The electron energy distribution in the plasma is affected by the metal being sputtered and the working gases' ionization and excitation potentials (EP). While keeping the magnetron power, rf coil power, target to substrate distance and pressure constant, the ionization fraction, as a function of position, has been measured. The electron temperature and density are measured using a Langmuir probe. The target metals analyzed in design of this experiment are aluminum(IP=5.98eV), copper(IP=7.72eV), and titanium(IP=6.82eV). The working gases will be krypton(IP=13.99eV, EP=1.702eV), argon(IP=15.76eV, EP=2.55eV), neon(IP=21.56eV, EP=3.52eV), and helium(IP=24.58eV, EP=5.36eV). An analytic model is compared to the experimental results.

9:40am **VM+TF-MoM5 Effects of Coil dc Potential on Ion Energy Distribution Measured by an Energy-resolved Mass Spectrometer in Ionized Physical Vapor Deposition**, ***E. Kusano***, *T. Kobayashi, N. Kikuchi, K. Fukushima, T. Saitoh, S. Saiki, H. Nanto, A. Kinbara,* Kanazawa Institute of Technology, Japan

In ionized physical vapor deposition, ion energy distribution is crucial to obtain films with desired properties. The energy distribution is supposed to be affected by the plasma potential that relates to the coil dc potential induced by an applied rf power. In this study, ion energy distribution of ionized Ti particles and Ar discharge gas has been measured by an energy-resolved mass spectrometer for various coil dc potential. The sputtering cathode used in the experiment was a conventional magnetron sputtering source with a Ti target (55mm@phi@). The cathode was coupled with an rf coil (60mm@phi@, made of Cu) generating an additional plasma in the region between the target and the substrate. The mass spectrometer was a Balzers PPM421 plasma monitor. The orifice to the ion optics was 0.1mm@phi@ and electrically grounded. The coil dc potential was controlled by changing the resistance of the resistor in the LCR circuit connecting the coil to the ground. The results showed that the energy of Ti@super +@ and Ar@super +@ was enhanced from a few eV to more than 100eV as a coil rf power increased from 0 to 200W for a constant cathode dc current. By changing the resistance of the LCR circuit, the peak of the energy spectra shifted from about 160eV for the resistance of 0@OMEGA@(the coil was grounded) to about 100eV for the resistance of 1k@OMEGA@. In addition, it was found that the total energy of Ti@super +@ or Ar@super +@ arriving to the spectrometer increased as the resistance decreased. The results suggest that the coil potential to the ground affect the plasma potential and thus the energy distribution of ions arriving to the electrically grounded substrate through the plasma sheath.

10:00am **VM+TF-MoM6 Modeling of Large Cluster Synthesis**, ***A. Hosseini-Tehrani***, *F.K. Urban III,* Florida International University

The original idea of the ionized cluster beam (ICB) thin film deposition technique was based upon producing, ionizing and accelerating beams of atoms clusters from vaporized material onto a substrate in a vacuum environment, using a supersonic jet source. Simulation of this process using classical nucleation theory and one dimensional gas flow equations will be presented. This approach is an extension of previous methods used for simulation of condensation of water vapor during supersonic expansion in nozzles and simulation mechanism of large clusters from vaporized solid materials. Zinc cluster sizes predicted by the model are in qualitative and quantitative agreement with our experimental results. Simulation results will be presented for different materials as well. Recently, other methods of synthesizing clusters and nanoparticles, using different types of cluster source, like magnetron sputtering mounted in a cooled chamber, have come under development. We are in the process of extending the model for the magnetron sputtering gas aggregation cluster source and will present new results for this process.

10:20am **VM+TF-MoM7 Combined Monte Carlo and Fluid Sputter Transport Model in an Ionized PVD System with Experimental Plasma Characterization**, ***D.R. Juliano***, *D.B. Hayden, M.M.C. Allain, D.N. Ruzic,* University of Illinois, Urbana

A code has been developed to model the transport of sputtered material in a modified industrial-scale magnetron. The device has a target diameter of 355 mm and was designed for 200 mm substrates. The chamber has been retrofitted with an auxilliary RF inductive plasma source located between the target and substrate. The source consists of a water-cooled copper coil immersed in the plasma, but with a diameter large enough to prevent shadowing of the substrate. The RF plasma, target sputter flux distribution, background gas conditions, and geometry are all inputs to the code. The plasma is characterized via a combination of a Langmuir probe apparatus and the results of a simple analytic model of the ICP system. A Monte Carlo routine in the code then tracks high energy atoms emerging from the target as they move through the chamber and undergo collisions with the electrons and background gas. The sputtered atoms are tracked by this routine whatever their electronic state (neutral, ion, excited). If the energy of a sputtered atom decreases to near-thermal levels, then it exits the Monte Carlo routine as is tracked with a simple diffusion model. In this way, all sputtered atoms are followed until they hit and stick to a surface, and the velocity distribution of the sputtered atom population (including state information) at each surface is calculated, especially the substrate. Through the use of this simulation the coil parameters and geometry can be tailored to maximize deposition rate and sputter flux uniformity.

10:40am **VM+TF-MoM8 Plasma Diagnostics of Magnetic Field Assisted Ionized Magnetron Sputtering**, ***J.H. Joo***, Kunsan National University, Korea, South Korea

The effects of axial magnetic field generated by solenoid coil has been studied for controlling the capacitive coupling between RFI antenna and metallic chamber wall, which causes severe coil sputtering at high RFI power levels. From OES results, at small magnetic flux density of 8G, RFI plasma showed sharp drop of plasma potential and reduced emission from Cu coil. Also visually the RFI plasma was confined within the RFI coil area. We compared two types of coil materials, metallic and ceramic coated. The plasma potential varied very much with materials and RFI power, which will affect the incomming ion's energy distribution. Also pulsing the sputtering power was studied to control average electron temperature of the RFI plasma, where electrons are easily quenched by heavily sputtered metals. As time dependent measuring of the plasma parameters is not readily available, some metallic films were deposited with different duties and the resulting film properties were measured. There was a big difference in preferred orientations of the grown Ag films. And the effects of ceramic coating on the RFI antenna will be addressed in the view point of plasma diagnostics, electron temperatures, electron densities, plasma potentials, contaminations and the change when it is coated by sputtered metals. Also the impedance characteristics of the RFI plasma were measured by RFZ-60 impedance analyser to study the type of coupling in mixed plasmas of DC magnetron and RFI plasma.

11:00am **VM+TF-MoM9 Study of Thin Films Deposited from a Copper Beam Formed in an Argon Atmosphere Capable of Condensing Nanoparticles**, ***F.K. Urban***, *A. Khabari, A. Housseini-Tehrani, P. Griffiths, G. Fernandez,* Florida International University

Although thin films formed from beams of nanoparticles or clusters have been discussed since the early 1970s, the question of the usefulness of this method has remained open as few films of any significant thickness have been formed to date. Early attempts did not condense, could only condense a few "high vapor pressure" solids, or were so low rate as to make growth too slow to be of use. A new deposition system has been designed and built here along the lines of those of Averback and Haberland, as they appear to have the most promise. The new system was specifically designed for high rate with a high throughput intermediate pressure pump and 2 kW capable sputter source. Preliminary films of copper deposited onto single crystal silicon substrates show a small beam divergence of less than 1 degree total. The beam is highly non-uniform with maximum intensity on-axis, which drops rapidly to zero within less than 10 mm off axis. Deposits have been made using a 1 Torr Argon + Helium sputtering and condensation atmosphere followed by nozzle aperture extraction. Films are affected by the amount of He and by cooling of the sputter chamber walls using liquid nitrogen. Nothing appears (detectable optically) on the substrate using Ar and no cooling and increases in both

factors result in films of generally increased thickness. Optically transparent films of copper have been deposited but are not yet understood. SEM, TEM, and AFM results of Cu and other films will be presented.

11:20am **VM+TF-MoM10 Novel (111)-Textured AlCu Growth by Ionized Metal Plasma (IMP) Ti Underlayer**, ***J.-B. Lai***, *L.-J. Chen,* National Tsing-Hua University, Republic of China*; C.-S. Liu,* Taiwan Semiconductor Manufacturing Company, Republic of China

(111)-textured AlCu is well known to possess better electromigration resistance than those of (200) and random orientations. In general, stronger (111)-textured AlCu can be obtained with the deposition of Ti underlayer compared with AlCu deposited directly on oxide or TiN underlayer. The improved texture is attributed to the small lattice mismatch between (0002)Ti and (111)AlCu. In this paper, (111)-textured AlCu (0.5 at. %) enhanced by Ti had been investigated using Auger electron spectroscopy, x-ray diffraction, transmission electron microscope, high-resolution transmission electron microscope, four-point-probe and EM test. Using thicker ion metal plasma (IMP) sputtered titanium underlayer was found to enhance the stronger growth of (111)-textured AlCu compared to conventional and collimated Ti films because of stronger (0002) textured-Ti was formed and hence the growth of (111)-textured AlCu was facilitated. However, TiAl@sub 3@, about 50 µm@OMEGA@-cm in resistivity, was found in the samples annealed at 400 °C. As the samples were annealed at 450 °C, a continuous but not smooth TiAl@sub 3@ precipitate layer was observed. If the precipitates of TiAl@sub 3@ were discontinuous and restricted to grain boundaries, the (111)-textured Al was destroyed and local joule heating caused the early failure of AlCu line. TiN, as a barrier layer, can retard the growth of TiAl@sub 3@. In our study, TiN/IMP-Ti was also found to enhance the stronger tendency of the growth of (111)-textured AlCu than TiN/collimated-Ti and TiN/conventional-Ti. The growth of (111)-textured TiN was enhanced by (0002)Ti. AlCu/TiN/IMP-Ti samples were observed to possess longer EM lifetime compared with those of AlCu/TiN/collimated-Ti (or conventional-Ti).

11:40am **VM+TF-MoM11 Plasma Polymerization of Fluorine Alloyed Amorphous Carbon Coatings**, ***A. Vanhulsel***, *J.-P. Celis,* KU Leuven, Belgium*; E. Dekempeneer, J. Smeets,* VITO, Belgium

This paper reports on the deposition conditions and characterization of plasma polymerized fluorocarbon coatings grown by an inductively coupled r.f. plasma (ICP) source, using CH@sub 4@ and CF@sub 4@ as precursor gases. SiH@sub 4@, H@sub 2@or Ar were further added to the plasma to investigate their influence on the coating properties. The coatings were characterized by XPS to determine the surface and bulk composition and combined with FTIR-spectroscopy to reveal the structure of the coatings. The mechanical properties (hardness and Young's modulus) were measured by nano-indentation. The surface energy was obtained by contact angle measurements with 2 different liquids. By varying the deposition conditions, we were able to deposit coatings with surface energies as low as 14 mN/m. With the appropriate feed gases and process parameters it is possible to adjust separately the polar and dispersive part of the surface energy. A low polar component of the surface energy corresponds to a high fluorine ,CF@sub 3@and CF@sub 2@ content at the surface of the coatings. The wetting behaviour of the coatings against water is mainly dependent on the polar component. The maximum contact angle achieved was 113°. By adding H@sub 2@ to the plasma, it is possible to minimize the polar component and maximize the dispersive component of the surface energy to obtain a relatively hard (3 GPa) coating with a hydrophobic nature (contact angle (H@sub 2@O) = 90°). The fluorocarbon coatings deposited in this system are not sensitive to atmospheric aging.

## Thin Films Division
## Room 310 - Session TF-MoA

### Mechanical Properties of Thin Films
**Moderator:** F.K. Urban, Florida International University

2:00pm **TF-MoA1 Computer Simulation Modeling of Sculptured Thin Films**, ***V.C. Venugopal***, *R. Messier,* Pennsylvania State University

Sculptured Thin Films (STFs) are characterized by free standing columns whose shape can be engineered as desired. Films with S-shaped, C-shaped, helicoidal or chevronic columns have been grown. Low adatom mobility and self-shadowing effects are critical for the growth of such films. To aid characterization of STFs theoretically, the growth of STFs is simulated using a simple ballistic aggregation deposition model assuming a high sticking coefficient, negligible relaxations, and low substrate temperatures. Substrate manipulations and complex substrate topographies are investigated. Clustering is found at the 1-3 nm level and is related to the larger column sizes which result from competitive growth evolution. A 3-D model of a growing STF is built up. The model structures are directly correlated to our experimental results in the relations between the incoming vapor angle @chi@@sub v@, and the resulting morphology column angle @chi@@sub m@. The final simulated structure is being used to develop mechanical models of STFs. Mechanical strength and elastic moduli can be determined at several different continuum levels and verified experimentally to develop a reliable model. Residual stress fields and other mechanical characteristics can also be studied. Using acoustic wave propagation principles, the maximum theoretical strength of these films can be determined and verified experimentally. Initial results of such models developed are presented.

2:20pm **TF-MoA2 Characterization of TiN/CN@sub x@ Multilayers Deposited by DC Magnetron Sputtering**, ***M.M. Lacerda***, *Y.H. Chen,* Northwestern University*; W.C. Chan,* University of Hong Kong, China*; B. Zhou, Y.W. Chung,* Northwestern University

Titanium nitride (TiN) is commonly used in wear protection coatings due to its high hardness. However, it is well known that thick TiN films develop columnar structure and are subject to cohesive failure. In this work, we used CN@sub x@ thin films to interrupt the growing TiN before the columns could initiate. The samples have been deposited by DC unbalanced magnetron sputtering at low pressure (2.5 mTorr) using an argon-20% nitrogen mixture. We applied a substrate bias of -200 V to promote ion bombardment. The CN@sub x@ thickness was kept constant at 1.3 nm. X-ray diffraction (XRD) patterns obtained at low angles (2°@<=@2@theta@@<=@5°) showed good interface between layers. XRD patterns at higher angles showed strong TiN (111) texture. The mechanical properties of the multilayers have been studied as a function of the TiN/CN@sub x@ thickness ratio (t@sub r@). Nanoindentation of samples with t@sub r@ = 2.3 showed high hardness value as compared to TiN films deposited at the same conditions. The TiN/CN@sub x@ films are at least 2.5 times harder than TiN samples. Internal stress of the same samples was calculated by the substrate curvature. Results showed that multilayers have compressive stress up to 10 times lower than TiN films. Electron microscopy results of the microstructure of these multilayered coatings will be presented.

2:40pm **TF-MoA3 Characterization of Stress-Morphology Relationships in Sculptured Thin Films (STFs)**, ***R.A. Knepper***, *D.E. Fahnline, R. Messier,* Pennsylvania State University

Sculptured Thin Films (STFs) are a recent development in thin film technology wherein a substrate is rotated while a columnar thin film is deposited at varying oblique angles and orientations. The resulting microstructure can thus be engineered into a number of shapes, including non-normal matchsticks, zigzags, coils, and periodically bent nematics (S- and C-shapes). STFs are highly porous and can have properties that differ greatly from both the bulk material and isotropic films of the same material. However, the nature of the relationships between STF morphology and intrinsic deposition stress is not yet understood. In this work, a set of matchstick-shaped STFs has been prepared with varying vapor incidence angles, ranging from 15° 75° from the substrate surface. The deposition results in curvature changes along the two directions of the substrate surface that have been measured by a laser scanning method. These measurements are then used to calculate the biaxial stresses in the films. The measured stresses decrease with decreasing morphology angle, with an abrupt change at a vapor incidence of 45°, and are anisotropic with respect to direction. The stresses measured ranged from 60 MPa to 0.4 MPa. The implications of these results to the general sets of STFs will be discussed. The findings of this research may be used to evaluate future models of the origins of stress in STFs, as well as to control substrate curvature and avoid delaminations.

3:00pm **TF-MoA4 Mechanical Properties Measurements using Scanning Force Microscopy**, ***W.N. Unertl***, University of Maine **INVITED**

Considerable effort is aimed at using the Scanning Force Microscope (SFM) to measure the mechanical properties of surfaces with nanometer-scale resolution. The properties of interest include the Young and shear moduli, shear strength, and work of adhesion. The most widely used approach is to extract these properties from the SFM data by simply scaling the results of macroscopic continuum contact mechanics theory to the dimensions and forces of an SFM contact. This talk will focus on two aspects of this scaling problem@footnote 1@: (1) the mechanism for failure of a contact under shear and (2) the effect of creep on contacts to viscoelastic materials. In a contact subjected to a shear strain, contact mechanics predicts that a crack propagates at the interface and causes a non-linear increase in shear force until the interface ruptures and sliding begins. This behavior, called microslip, is observed for macroscopic contacts but not for SFM contacts, which suggests that the contact mechanics picture must be modified for nanometer-scale contacts. In contacts to viscoelastic materials, creep can significantly modify the formation and rupture of a contact compared to contacts to elastic materials. The most important effect is that the maximum contact area depends on the loading history and, unlike elastic materials, can reach its maximum value well after the maximum load is applied. The status of theoretical models for the analysis of contacts to linear viscoelastic solids including the effects of adhesion will be described. @FootnoteText@ @footnote 1@K.J. Wahl, S.V Stepnowski, W.N. Unertl, Tribology Lett. (in press 1998).

3:40pm **TF-MoA6 Meso-Scale Contact Hardness, Friction, and Wear of Aluminum Oxynitride Films**, ***S.D. Dvorak***, *G.P. Bernhardt, O.D. Greenwood, R.J. Lad,* University of Maine

Aluminum oxynitride (AlO@sub x@N@sub y@) thin films attract interest as hard, wear resistant coatings for high temperature, oxidizing environments. We have used electron-cyclotron-resonance (ECR) plasma assisted electron beam evaporation of aluminum to grow aligned crystalline films on r-sapphire at 800 - 1100 K to nominal thicknesses of 100 nm at about 0.5 Å/s deposition rates. These AlO@sub x@N@sub y@ films were fabricated with compositions ranging from aluminum oxide to aluminum nitride, depending on the N@sub 2@/O@sub 2@ gas flow ratio in the ECR source. Film hardness as a function of depth was measured by nanoindentation, while friction and wear properties were determined during reciprocal sliding experiments using well characterized sapphire and diamond probe tips with applied loads in the micro-newton to milli-newton force range. Film topography examined with atomic force microscopy indicated rms roughness values ranging from 20 Å to 140 nm. Wear tracks examined by AFM consist of wear debris as well as microstructural features. We observe that friction is affected by the roughness of the surfaces in contact, and that these roughness effects are dependent on the hardness of the contacting asperities, as measured by a Hysitron Pico-Indentor. Inhanced surface diffusivity of oxygen and nitrogen species provided by the ECR source during film growth yields highly-oriented films with very high wear resistance.

4:00pm **TF-MoA7 Nanotribology of Single Crystal ZnO Surfaces: Relation of Atomic Level Friction to Macro Tribology of Thin Films**, ***J.J. Nainaparampil***, *J.S. Zabinski, S.V. Prasad,* Air Force Research Laboratory

Atomic Force Microscopy (AFM) has been applied to the study of surface forces for more than a decade. Relatively recently, Lateral Force Microscopy (LFM) has evolved from AFM as a means to characterize surface forces in relation to friction, adhesion and surface topography. The significance of this approach is that it reveals insights into friction and wear at an atomic level. This work focuses on the nanotribology of single crystal ZnO surfaces after high temperature annealing treatments and in different gases. Annealing causes the formation of surface structures - etch pits on the 1010 surface and roughening or reconstruction on the 0001 surface. The pits and roughened areas provided lateral force contrast that could not be assigned to topography. Adhesion and relative contact stiffness were not significantly different among friction contrasting regions. The chemistry of these regions was analyzed using Scanning Electron Microscopy (SEM) and Scanning Auger Microscopy (SAM). The LFM and chemical analysis of the different single crystal surfaces will be presented. Insights into atomic

level friction and wear processes will be related to the macroscopic tribology of ZnO nanocrystalline thin films.

4:20pm **TF-MoA8 Surface Stress in Silicon Oxide Layer made by Plasma Oxidation with Applying Sample Bias**, *A.N. Itakura,* National Research Institute for Metals, Japan*; T. Kurashina, T. Narushima,* University of Tsukuba, Japan*;* ***M. Kitajima,*** National Research Institute for Metals, Japan

We present the evolution of surface stress during plasma oxidation of Si(100) with applying bias voltage from -60V to +60V to the sample. The experiments were performed in a UHV condition. Oxide thickness was controlled from 0 to 3nm. The sample was a cantilever of Si(100) of dimensions 450µm x 50µm x 4µm. The bending of the lever due to stress was detected as a function of the oxidation time by a change in the reflection angle of laser beam from lever backside. Stress was calculated from the lever deflection using Stony's formula. The plasma was generated by RF discharge of oxygen gas at 13.56MHz. There has been observed three stages in the stress vs. time curve for the plasma oxidation of Si cantilever without applying sample bias. The first stage was rapidly building up of tensile stress and the second stage was the tensile stress decreasing slowly. In the last stage the stress changed to compressive. For the cases of oxidation with applying bias to the samples, stress curves showed different time dependence from that without bias. First, the stress curve showed a quick build-up of compressive stress, followed by a tensile stress formation, and the stress gradually changed to compressive one with further oxidation. The similar feature appeared in the curves with positive biases and negative biases. The stress values were not unique at same thickness but strongly depended on a bias voltage. We will discuss these stress changes in terms of the interface structure of silicon-oxide layer and silicon substrate.

4:40pm **TF-MoA9 Stress Alignment in SiO@sub 2@ Thin Films Deposited on Thin Chromium and Aluminum Film**, ***K.E. Coulter***, *V. Raksha,* Flex Products, Inc.

SiO@sub2@ as a low index material in optical applications is often complicated by the intrinsic stress that induces film cracking, substrate deformation and delamination. 400 - 600nm thick SiO@sub2@ films were deposited by e-beam evaporation onto Cr (10nm) and Al (100nm) films. Using design of experiment methodology, we evaluated deposition process parameters such as rate, vacuum pressure, substrate type, coating material and storage conditions. The thin films were deposited on 50 and 175 µm thick PET substrates as well as fused silica witnesses. Analysis methods included interferometry, profilometry, microscopy, ellipsometry and an evaluation principle based on laser deflection off the free end of a coated PET strip. A correlation was established between absolute stress values measured by interferometry and the laser deflection method. Cr/ SiO@sub2@ and Al/ SiO@sub2@ films were deposited under vacuum conditions which produced tensile and compressive stress. S! tress in all SiO@sub2@ thin film stacks became more tensile with age (shelf life) regardless of the initial stress in the film. Dopants in the silica source material such as B@sub 2@O@sub 3@, Na@sub 2@O and Vycor produced films with similar correlations between stress and deposition conditions but at lower absolute stress magnitudes. In this presentation, the influence of the vacuum pressure, deposition rate, storage conditions and substrate properties will be discussed relative to the effect of stress on the thin film optical performance.

5:00pm **TF-MoA10 Investigation of Induced Recrystallization and Stress in Close-Spaced Sublimation CdTe Thin Films**, ***H.R. Moutinho***, *R.G. Dhere, M.M. Al-Jassim, P. Sheldon,* National Renewable Energy Laboratory*; B.T. Mayo,* Southern University*; L.L. Kazmerski,* National Renewable Energy Laboratory

Close-spaced-sublimation (CSS) CdTe has produced the best CdS/CdTe thin-film solar cells reported to date. In all CdTe cell deposition options, a post-deposition treatment with CdCl@sub 2@/methanol solution at elevated temperature is a mandatory step for maximizing the device efficiency. We have previously reported that these large-grain CSS films do not recrystallize and that the initial in-plane stress is not completely relieved during the treatment, in contrast to films deposited by other methods (e.g. physical vapor deposition). In this work, we deposited CSS CdTe films at lower temperatures and higher deposition rates to force lower-grain-size layers, which are more susceptible to recrystallization. The objective was to induce recrystallization from the chemical/heat treatment to realize films with substantially less stress and, consequently, better device quality. The CdTe films were deposited on normal CdS/SnO@sub 2@/glass structures and chemically treated at various temperatures and times to optimize the recrystallization process. The topography and grain size of the films were determined by atomic-force microscopy, X-ray diffraction, and transmission electron microscopy, and the minority-carrier lifetime by time-resolved photoluminescence. The CdCl@sub 2@ treatment temperature was varied from 300 to 400° and the treatment time from 1 to 30 minutes. The stress in the films was investigated using X-ray techniques, and significant reduction in the stress was observed concurrent with the recrystallization/recovery process. We investigated the evolution of stress in the early stages of the treatment to establish the mechanisms through which recrystallization starts in these films. CdTe films deposited by physical vapor deposition were also analyzed, and the results were compared with the ones for the CSS films. Finally, the efficiencies of cells prepared from conventional CSS CdTe were compared to these lower stress thin-film devices.

## Thin Films Division
## Room Hall A - Session TF-MoP
## Thin Films Poster Session

**TF-MoP1 Studies on Anti-Glare, Anti-Static and Transparent Conductive Film on Display Tubes**, ***G.K. Xi***, *G.H. Zhang, S.L. Li, S.M. Shao, W. Guo, X.J. He,* Nankai University, China

Anti-glare,anti-static thin film and transparent conductive film are widely used in many fields of production, science and technology, such as solar cell,TV image display panel, instrument panel and transparent electrode. Anti-glare and anti-static double-layered coating on ordinary glass prepared by a sol-gel technique is introduced. The outer layer of low refractive index is coated on high refractive index inner layer. The main composition of outer layer is SiO@sub 2@. Some SnCl@sub 4@ added into this film increases the conductivity of this film. The latter can be replaced by other hygroscopic metal salts,such as Al(NO@sub 3@) @sub 3@,AlCl@sub 3@,ZnCl@sub 2@, etc.. The inner layer is composed of TiO@sub 2@ and SiO@sub 2@. The glass panel with the double-layered coating shows the resistivity of 10@super 9@ @OMEGA@/square and reflectance of 1.6 % which is reduced to one-fourth of that without coating. A transparent film with high conductivity on ordinary glass panel prepared by a sol-gel technique is also introduced. This Sb-doped SnO@sub 2@ film is made from alkoxide which was previously prepared in our lab. This ATO film has a polycrystall structure. The resistivity of the ATO film has minimum value of 10@super 3@ @OMEGA@/square while the doping level of the solution is about 7%. Finally,factors such as enviromental temperature and humidity,coating technology,effecting the characteristics of these films are discussed.

**TF-MoP2 Humidity Sensing Properties of Plasma Polymerized Organic Thin Films**, ***G.B. Park***, Yuhan College, Korea*; J.T. Kim, D.C. Lee,* Inha University, Korea*; C. Kim,* Korea Electronic Technology Institute

In order to fabricate humidity sensitive films, the mono-layer polymer thin films from various monomers were deposited on a comb-shaped electrode using a capacitive-coupled gas flow type plasma polymerization apparatus with rf power. The humidity sensing characteristics of these thin films were measured by means of changes in electrostatic capacitance within the frequency and humidity range of 60Hz - 100 kHz and 20% RH - 90% RH, respectively. The capacitances of these thin films were increased with increasing relative humidity. And the increments of the capacitance grew larger with a lower discharging power and a shorter polymerization time. The linearity and increment of the capacitance change became apparent with the lower frequency of input power to thin films during the capacitance measurements. The copolymerized thin films, vinylacetate and methylmethacrylate, were fabricated using the conditions of low discharging power and short discharging time. The capacitances of this films were remarkably changed showing the linear behaviors, and these changes were continued from 20% RH to 90% RH.

**TF-MoP3 Rotating-Compensator Spectroscopic Ellipsometry: Applications of Four-Parameter Stokes Vector Spectroscopy to Real Time Characterization of Non-Ideal Thin Films**, ***R.W. Collins***, *P.I. Rovira, J.C. Lee,* Pennsylvania State University

We have developed a multichannel ellipsometer in the rotating-compensator optical configuration, i.e., (fixed polarizer)-(sample)-(rotating compensator)-(fixed analyzer). This instrument has the advantage of providing the spectrum in the degree of polarization p of the light beam reflected from the sample, in addition to the spectra in the ellipsometric angles (@psi@, @DELTA@). In a recent advance, we have extended the instrument capabilities to a simultaneous measurement of the spectral reflectance R of the sample. As a result, the new instrument can collect four-parameter spectra [(@psi@, @DELTA@), p, R] that characterize the unnormalized Stokes vector of the reflected light beam. The minimum measurement time is 32 ms for all four spectra from 1.5 to 4.0 eV. In this paper, instrumentation and calibration issues specific to the simultaneous reflectance measurement will be described. Applications of the rotating-compensator instrument to date include (i) optical anisotropy in nanoscale sculptured thin films of MgF@sub 2@, (ii) nucleation and growth of nanocrystalline and polycrystalline diamond films, and (iii) optical properties, structure and stability of specular and textured transparent conducting oxide thin films. We review these applications and highlight the unique capabilities developed so far. These include the use of p along with (@psi@, @DELTA@) to characterize the evolution of thickness non-uniformity during the growth of diamond films, and the use of R along with (@psi@, @DELTA@) to characterize the effect of annealing and H@sub 2@-plasma exposure on the optical properties and surface roughness on micro/macroscopic scales for textured SnO@sub 2@:F used in photovoltaics applications.

**TF-MoP4 Characterization of Thin Metal Films Processed at Different Temperatures**, ***L. He***, *J.E. Siewenie,* Northern Illinois University

Thin metal films are of considerable interest for electronic device fabrication. Not only do these films provide electrical interconnection between circuit elements, they can also be an integral part of a circuit element, as in the case of Schottky diodes and metal semiconductor field-effect transistors(MESFETs) and metal-semiconductor-metal photodetectors (MSM PDs). It is well known that the electrical conduction in metals is due to electrons, while electrical resistivity, defined as the reciprocal of the conductivity, is the result of electron collisions. The high resistivity of thin metal films result in drawback of their applications in afore mentioned devices. Recent studies have been conducted in thin metal films obtained by low temperature (LT = 77K) deposition. Comparing to the same film formed at room temperature, the LT film resistivity could be 4 to 5 orders lower in magnitude. In another hand, metal/semiconductor Schottky barrier heights are significantly increased in materials including InP, GaAs, and InGaAs. This work extensively studied the electrical and micro-structural properties of several often used metal films including Au, Ag, Al, Pt, Pd, and Ni formed at LT and RT. Atomic force microscopy (AFM), transmission electron microscopy (TEM), and in-situ resistivity measurements were conducted. In AFM surface scanning, surface morphology consistently showed the LT and RT films difference in the grain sizes. The larger grain size in LT thin film explains the lower resistivity. TEM electron diffraction pattern showed very different degree of crystallization of the LT and RT thin films. The LT film diffraction pattern suggests that LT films develop a less regular structure which may be responsible for the Schottky barrier enhancement. LT deposition prevents both adatom diffusion and re-evaporation, leading to the earlier conducting than the same RT films.

**TF-MoP5 Optical Properties of the Ge:Sb:Te System**, *E. Garcia-Garcia,* Univ. Autonoma de Queretaro, Mexico*; A. Mendoza, G. Martinez-Montes,* Univ. Autonoma de Puebla, Mexico*; Y.V. Vorobiev,* ***J. Gonzalez-Hernandez***, CINVESTAV-IPN, Mexico*; B.S. Chao,* Energy Conversion Devices

Stoichiometric compositions of the Ge:Sb:Te system are commercially used for optical data storage. In this work, we have measured using ellipsometry, the optical constants (n, k) in the range of 1.4 to 6 eV in all the stoichiometric composition in the Ge:Sb:Te system. It is known that the amorphous phase of these compositions undergoes an amorphous-to-crystalline transition at temperatures in the range from 140-160 °C depending on Sb concentration. The crystalline structure of this phase is the fcc. Heat treatments at temperatures above the mentioned range produce a new crystalline-to-crystalline transition from the fcc to the hexagonal phase. The n values for the amorphous and crystalline fcc phase are not so different and both decrease from a value of about 5 to 1.4 eV to a value of 1 at 6 eV. In general, this phase transition produces an increase in the values of k in the whole energy range. The observed changes in n and k in the hexagonal phase are more complex due to the increase in the free charge density and will be discussed in the extended presentation.

**TF-MoP6 Influence of Annealing Temperature on the Formation and Characteristics of Sol-gel Prepared ZnO Films**, *R. Castanedo-Pérez, O. Jiménez-Sandoval, S.J. Jiménez-Sandoval, A. Maldonado-Alvarez, J. Márquez-Marín,* ***G. Torres-Delgado***, Cinvestav-IPN, Mexico

ZnO films have been obtained by the sol-gel method, from a Zn(OOCCH@sub 3@) precursor, on silica glass and silicon wafer substrates. The films, obtained by a single dipping procedure, were characterized by FT-IR and UV-VIS spectroscopy, atomic force microscopy, X-ray diffraction and ellipsometry measurements. Untreated and single-step, annealed (100-450°C) films were studied, in order to analyze the influence of temperature on the formation and properties of the ZnO coatings. Remarkably, these results indicate that ZnO forms at considerably lower temperatures than 450°C, which is usually considered in literature as a reference temperature for the formation of ZnO. Thus, a sharp absorption edge of ZnO at ca. 380 nm, can be neatly observed in the UV-VIS spectra of films annealed at 200 and 300°C, and accordingly, IR data indicate the absence of organic groups at these temperatures. Somewhat surprisingly, at 400 and 450 °C, the ZnO optical absorption edge is not as sharp as is at lower temperatures. Atomic Force Microscopy results show larger grain sizes as the annealing temperature is increased. The X-ray diffraction

patterns show that the films are polycrystalline and also evidence the formation of ZnO at temperatures as low as 200°C.

**TF-MoP7 Structural Characterization of SrBi@sub 2@Ta@sub 2@O@sub 9@ Ferroelectric Thin Films Grown by PLD on Pt and RuO@sub 2@ Bottom Electrodes**, ***J.M. Siqueiros***, UNAM, Mexico; *M.P. Cruz,* CICESE, Mexico; *J. Portelles,* Universidad de la Habana, Cuba; *R. Machorro, G. Hirata, S. Wang,* UNAM, Mexico

To study the effect of the bottom electrode on the properties of the ferroelectric layer, thin SBT (SrBi@sub 2@Ta@sub 2@O@sub 9@) films were deposited by PLD on Pt/Ti/Si, Pt/TiO@sub 2@/Si and RuO@sub 2@/Si grown by DC sputtering. Due to the previously reported experience that the Ti in the adherence promoter layer diffuses to the Pt surface, a study of the SBT/electrode interface using AES and TEM is performed. XRD, SEM, STM and ellipsometry measurements were performed on the SBT films and compared with the corresponding measurements on the ceramic used as target for the deposit, which showed a layered perovskite structure.

**TF-MoP8 Characteristics of CuInSe@sub 2@ Thin Films Prepared in Different Selenization Pressures**, ***S.D. Kim***, *C.H. Chung,* Seoul National University, Korea; *K.H. Yoon, J.S. Song,* Korea Institute of Energy Research, Korea; *H.J. Kim,* Seoul National University, Korea

CuInSe@sub 2@ based solar cell has great interest because CuInSe@sub 2@ has high absorption coefficient, suitable bandgap energy, good thermal stability, and good lattice match with window layers such as CdS, Cd(Zn)S. Its conversion efficiency has been reached above 15%. The selenization method has been known as an excellent technique to acquire low cost and high efficiency CuInSe@sub 2@ thin films. The effects of chamber pressure during selenization of Cu-In alloy layers on the optical, electrical and structural properties of CuInSe@sub 2@ films were investigated. The uniform Cu-In alloy layers could be reproducibly deposited on the glass substrate by dc co-sputtering method. The two atmospheres, Ar atmosphere at 1 atm and in vacuum of 10 mTorr, were chosen for the selenization of Cu-In layers. The properties of all films were analyzed by XRD, SEM, EDX, four point probe, Raman spectroscopy and photoluminescence. Cu-In precursors consisted of two phases, CuIn@sub 2@ and Cu@sub 11@In@sub 9@, and the amount of Cu@sub 11@In@sub 9@ phase increased with varying the composition from In-rich to Cu-rich. Less compounds of Cu-Se and In-Se were observed during the early stage of selenization and also CuInSe@sub 2@ single phase was more easily formed in vacuum than at atmospheric pressure. Therefore, CuInSe@sub 2@ films selenized in vacuum showed large grain size, smooth surface, dense microstructure, high Raman peak intensity and no secondary phases with near-stoichiometric composition. Since CuInSe@sub 2@ films selenized in vacuum could hardly release the intrinsic stress due to dense structure, Raman peak of 173 cm@super -1@, A1 mode of CuInSe@sub 2@ charcopyrite phase, shifted to higher frequency and had a broad full width of half maximum.

**TF-MoP9 Growth and Characterization of Epitaxial Films of Tungsten-Doped Vanadium Oxides on Sapphire (110) by Reactive Magnetron Sputtering**, ***P. Jin***, *M. Tazawa, M. Ikeyama, S. Tanemura,* National Industrial Research Institute of Nagoya, Japan; *K. Macak, X. Wang, U. Helmersson,* Linkoping University, Sweden

Some vanadium oxides undergo a semiconductor-to-metal phase transition with significant changes in optical, electrical and magnetic properties. Replacement of V by metals such as W, Mo affects greatly the properties. Since thin films of such materials are candidates for switching or memory devices, it is necessary to investigate the growth, structure and properties of the metal-doped vanadium oxides particularly in the form of epitaxial film. In this study, films of W-doped vanadium oxides were epitaxially grown on sapphire (110) by reactive sputtering a V-W alloy target. With computer control of deposition parameters, especially the oxygen flow into the Ar+O@sub 2@ discharge, a series of epitaxial films having structures of not only the best known MO@sub 2@ (M=V+W) but also others from M@sub 2@O@sub 3@ to M@sub 2@O@sub 5@ were obtained. The films were studied with X-ray diffraction (XRD), atomic force microscopy (AFM), Rutherford backscattering spectrometry (RBS), etc. The XRD theta-2theta scan shows only pairs of peaks corresponding to those from the film and the substrate. Furthermore, a series of films formed by varying slightly the oxygen flow exhibit a continuous shift of the XRD peaks in response to their metal-to-oxygen ratio. In other words, a continuous structural change was obtained by precise process controlling. The XRD pole figure study confirmed the epitaxial relationships. The phase transition properties were studied from the change in electrical resistivity against temperature. The results demonstrate strong influences both from the metal-to-oxygen ratio and the tungsten doping amount.

**TF-MoP10 Stability of Transparent Conducting Oxide Films for Use at High Temperatures**, *T. Minami, T. Miyata,* ***T. Yamamoto***, Kanazawa Institute of Technology, Japan

The stability of transparent conducting oxide (TCO) films in various atmospheres at high temperatures is important for applications such as transparent heaters and also in optoelectronic device fabrication processes. However, the electrical, optical and chemical properties of TCO films at high temperatures above 500 ° C have not been investigated because of the melting point of glass substrates. In this paper, we describe the stability of various TCO films at high temperatures in various atmospheres. Various TCO films consisting of binary compounds such as ZnO, In@sub 2@O@sub 3@ and SnO@sub 2@, ternary compounds such as Zn@sub 2@In@sub 2@O@sub 5@, In@sub 4@Sn@sub 3@O@sub 12@, GaInO@sub 3@, ZnSnO@sub 3@ and MgIn@sub 2@O@sub 4@ or multicomponent oxides composed of two binary compounds or two ternary compounds were tested in this work. The TCO films were deposited by magnetron sputtering on quartz substrates at room temperature or 350 ° C. The tests were carried out in various atmospheres such as air, argon gas and vacuum at temperatures up to 1000 ° C. The SnO@sub 2@, In@sub 2@O@sub 3@ and In@sub 4@Sn@sub 3@O@sub12@ thin films were found to be more stable than other materials. It can be concluded that stability at high temperatures was mainly determined by the metal elements contained in the TCO film; high stability was obtained in TCO films rich in Sn and In.

**TF-MoP11 Texture and Grain Size Modification through the Different Sintering Conditions for Ceramics of the Sr@sub 0.5@Ba@sub 0.5@TiO@sub 3@ Type**, ***J.M. Siqueiros***, UNAM, Mexico; *J. Portelles, S. Garcia,* Universidad de la Habana, Cuba; *S. Aguilera,* Universidad de Catolica de Norte, Chile; *M. Xiao,* UNAM, Mexico; *A. Fundora,* Universidad de la Habana, Cuba

The variation of the sintering conditions: temperature and sintering time, strongly modify the texture and grain size of the SBT ferroelectric compound (Sr@sub 0.5@Ba@sub 0.5@TiO@sub 3@). In this report, the sintering time is varied from 1 to 5 hours. The grain size increases as the sintering temperature is varied from 1200 to 1450 @super o@C. SEM studies allow us to determine the corresponding microstructure for each temperature and sintering time. A linear dependence of logarithm of the grain size with the sintering temperature is found. A change in the dielectric permittivity and the polarization with the sintering conditions is also reported. The analysis of the results is supported with XRD, thermoelectric and dielectric hysteresis measurements

**TF-MoP12 Microstructure Study of PMN-PT Films Grown on Metal Electrodes by PLD**, ***J.M. Siqueiros***, UNAM, Mexico; *J. Portelles, A. Fundora,* Universidad de la Habana, Cuba; *S. Aguilera,* Universidad de Catolica de Norte, Chile

Thin ferroelectric films obtained by PLD from non-stoichiometric Pb(Mg@sub 1/3@Nb@sub 2/3@)O@sub 3@-PbTiO@sub 3@ (PMN-PT) ceramic targets are studied. The morphology of the resulting films for the 2.7PMN-0.1PT composition obtained in N@sub 2@ and O@sub 2@ atmospheres at room temperature obtained by SEM is reported. It is observed that, for similar deposit conditions, thicker films with columnar structure are produced in the O@sub 2@ atmosphere as compared with those grown on N@sub 2@ where very low crystallinity was detected. The film composition was determined by Auger electron spectroscopy and XPS. TEM measurements of the N@sub 2@ grown films showed scattered nanostructures embedded in a dominion structure in the paraelectric state, since the measurements were performed at room temperature, above the Curie temperature of the ceramic (15 @degree@C). This result seems to imply a diffuse phase transition associated to the film. Regions of high concentration of Niobium were detected by XPS evidencing the presence of pyrochlores, a situation confirmed by XRD. The PMN-PT/electrode interface is analyzed by TEM for samples annealed at different temperatures and the results are correlated with those obtained by XRD and SEM.

**TF-MoP13 Columnar Growth of Tin from Liquid Metal Ion Source Studied by In-situ Transmission Electron Microscopy**, ***H. Kimata***, *Y. Kondo,* ERATO, Japan Science and Tech. Corp., Japan; *K. Takayanagi,* Tokyo Institute of Technology, Japan

A miniaturized liquid metal ion source (LMIS) built in a conventional transmission electron microscope (TEM) was developed to observe field desorption of tin ions and droplets, and growth of deposit from them on

substrates in-situ. The LMIS has a reservoir, a needle and a filament which heats the liquid tin in the reservoir. The needle is faced to an extraction electrode with a small hole. A shield plate, with a small hole, is placed behind the extraction electrode. The substrate is placed behind the shield plate. Potentials of the LMIS and the extraction electrode are ground and negatively high, respectively. The shield plate and the substrate are usually ground. Thus emitted ions are decelerated to have near zero energy at the substrate, and they land the substrate very softly. Positive bias is occasionally applied to the shield plate and the substrate, to repel positive ions for selecting neutrals from mixture of ions and neutrals. Tin ions and neutrals were emitted from the LMIS at extraction voltage ranged from 4 to 6kV, with an emission current of about 40x10@super -6@A. We observed growth of tin on a substrate in-situ. The substrate was an amorphous carbon film on a thin tungsten wire. We found that columnar tin grew; typically 500nm in length and 50nm in width, when the substrate and the shield plate were ground. Columnar growth was not found when +100V bias was applied to the substrate and the shield plate, to repel the ions. The experiments showed that ions are essential for the columnar growth. The strong magnetic field (~2T) of the objective lens in the TEM, might play a role for the growth.

**TF-MoP14 Characterization of TiOx Film deposited on Ti-6Al-4V Alloy by Reactive Sputtering in Oxygen Atmosphere**, ***T. Sonoda***, *M. Kato,* National Industrial Research Institute of Nagoya, Japan

Coating of Ti-6Al-4V alloy substrates with TiOx films by reactive sputtering in oxygen atmosphere was examined, not only to improve the biocompatibility of the alloy@footnote 1@ but also to enhance the bonding of the alloy implants to living bone.@footnote 2@ The reactive sputter deposition was carried out in oxygen gas using a magnetron d.c. sputtering apparatus with a pure titanium target. Thus the TiOx films were deposited on the alloy substrates at the rate of 300Å/min by the magnetron sputtering, even under the surface condition at the target to be in the reactive mode,@footnote 3@ due to reactive products covering the surface. The characteristics of the deposited TiOx films were investigated. Under visual observation, the deposited films looked light gray and appeared to be uniform and adhesive. Under SEM, the surface of the films was found to have a net-like microstructure which looked like a net consisting of micron-ordered fine meshes. Under AES, the Ti/O ratio of each film was constant in depth direction of the film. Based on XRD, it was concluded that not only the oxides(TiO@sub 2@) such as rutil and brookite but also the suboxides such as Ti@sub 4@O@sub 7@ and Ti@sub 6@O@sub 11@ were formed in the film. Therefore, it was assumed that the obtained TiOx films improved the biocompatibility of the alloy and enhanced the bonding of the alloy to living bone. Furthermore, the hardness of the TiOx films reached over Hv=800 under the film thickness of 0.8μm, concluding that the coating with the films improved the hardness of the alloy. @FootnoteText@ @footnote 1@A.Wisby et al., Biomaterials 12(1991)470. @footnote 2@T.Kitsugi et al., J. Biomed. Mater. Res. 32(1996)149. @footnote 3@S.Schiller et al., Thin Solid Films 111(1984)259.

**TF-MoP15 Photoconductivity of Free-standing Diamond Film**, *S.-H. Kim,* Silla University, South Korea; *I.T. Han,* Samsung Advanced Institute of Technology, South Korea; ***T.-G. Kim,*** Miryang National University, South Korea

Thick diamond film having 700 μm thickness was deposited on polycrystalline molybdenum (Mo) substrate using high power (4 kW) microwave plasma-enhanced chemical vapor deposition (MPECVD) system. We could achieve free-standing diamond film by detaching as-deposited diamond film from the substrate. Parallel-type diamond photoconductors were fabricated on either the growth side or the susbtrate side of free-standing diamond film via ohmic contact metallization. We investigated the variation of photoconductivity after exposing the film surface to either oxygen or hydrogen plasma. At as-grown state, the growth side showed noticeable photoconductivity, while the substrate side gave little photoconductivity, The oxygen plasma treatment of these sides led to the insulators. After exposing the film surfaces to hydrogen plasma, on the other hand, we could observe not only distinct photoconductivity at the substrate side but also the reappearing of photoconductivity at the growth side. Finally, we suggest that the dangling hydrogen bond on the film surface may play an important role to create the photoconductivity of this film surface.

**TF-MoP16 Target Compound Layer Formation during Reactive Sputtering**, ***L.B. Jonsson***, *T. Nyberg, S. Berg,* Uppsala University, Sweden

It is well known that a compound layer may form at the target surface during reactive sputtering. However, the significance of this layer for the response to a change in target conditions has so far not been carefully investigated. The standard model for the reactive sputtering process @footnote 1@ does not allow for calculations of the compound thickness at the target surface. For simplicity it has been assumed that a single monolayer is responsible for the poisoning of the target. However, experiments clearly indicate that the compound layer thickness may be significantly thicker than one monolayer. For several reasons it is important to be able to quantify the thickness of this layer. The formation of the compound layer introduces memory effects into the system when the processing conditions are changed. The delay time for sputter erosion of the compound layer depends strongly on the thickness of the layer. We will present an extension of the basic reactive sputtering model that explains the formation of an arbitrary thickness of the compound layer at the target surface. From this model it is possible to examine the layer thickness dependence on the major processing parameters (reactive gas supply, sputtering power etc.). Optical emission spectroscopy studies of sputtered target material and transient target voltage response measurements confirm the validity of the new model. @FootnoteText@ @footnote 1@ S. Berg, H.-O. Blom, T. Larsson, and C. Nender, J. Vac. Sci Technol. A 5, 202-207 (1987).

**TF-MoP17 Characterization of YSZ(Yttria-Stabilized Zirconia) Thin Films Prepared by RF-Magnetron Sputtering for Oxygen Gas Sensor**, ***J.W. Bae,*** *J.Y. Park, G.Y. Yeom,* Sungkyunkwan University, Korea; *K.D. Kim,* Korea Gas Corporation, KOREA; *Y.A. Cho, J.S. Jeon, D.S. Choi,* Korea Gas Corporation, Korea

Yttria-stabilized zirconia(YSZ), a well known oxygen ion conductor, is one of the many solid state ionic materials utilized in the variety of electrochemical devices including fuel cells, oxygen pumps, and chemical gas sensors. Commercial YSZ oxygen gas sensors rely on the traditional bulk ceramic fabrication and require temperatures above 600 @degree@C to achieve sufficient ionic conductivity. However, if the YSZ could be applied as a thin film, it would offer many advantages including compact dimensions with smaller power consumption, reduced ohmic losses, and lower operating temperatures. In this experiment, yttria-stabilized zirconia(YSZ) films(0.5-1.5μm) were deposited on Pt/NiO-Ni mixed reference layer/SiO@sub 2@ substrates to characterize films properties using an RF-magnetron sputter deposition system and zirconia stabilized with 8mol% yttria(Y@sub 2@O@sub 3@) was used as a sputter target. The plasma atmospheres were pure Ar or mixtures of Ar and O@sub 2@. We employed X-ray diffraction to study the structure of YSZ films and scanning electron microscopy(SEM) to examine the film surface morphologies. Analyses by Auger electron spectroscopy(AES) and X-ray photoelectron spectroscopy(XPS) were performed to examine Zr, Y, and oxygen compositions in YSZ thin films and the uniformity in compositions. Transmission electron microscopy(TEM) was employed to examine the microstructural details and crystallography of the films. Gas-sensing test was carried out for Pt/YSZ/Pt/NiO-Ni/SiO@sub 2@ film structures exposed to an atmosphere of oxygen controlled composition. The deposition rates of YSZ thin films increased with increasing rf power and total pressure, and decreased with increasing O@sub 2@ concentration. The preferred orientation of deposited YSZ films changed from (111) to (220) with increasing thickness. O/Zr ratio analyzed by XPS was increased from 1.94 to 2.4 with increasing O@sub 2@/(Ar+O@sub 2@) ratio. But yttria atomic percentage in the YSZ films were not changed. More detailed physical and chemical characteristics of YSZ thin films and their relations to gas-sensing properties will be discussed in the presentation.

**TF-MoP18 Growth Characteristics and Deposition Mechanism of SrTiO@sub 3@ Thin Films by Plasma Enhanced MOCVD**, *Y-.B. Hahn,* ***D.O. Kim***, *K.S. Nahm,* Chonbuk National University, Korea

Dielectric SrTiO@sub 3@ ultra thin films having 30 - 75 nm thickness were deposited on Pt/Si and Ir/Si substrates by plasma enhanced MOCVD using high purity Ti(O-i-C@sub 3@H@sub 7@)@sub 4@, Sr(tmhd)@sub 2@ and O@sub 2@. Depositions were carried out under various operation conditions. The deposition rates were substantially influenced by bubbler temperature (T@sub b@), substrate temperature (T@sub s@) and rf power. The optimum conditions of deposition were T@sub b@ = 60 @super o@C for Ti(O-i-C@sub 3@H@sub 7@)@sub 4@ and 220 @super o@C for Sr(tmhd)@sub 2@, T@sub s@ = 550 @super o@C, 130 sccm O@sub 2@, and 160 W rf with carrier gas flow rates of 40 sccm for Ti(O-i-C@sub 3@H@sub 7@)@sub 4@ and 130 sccm for Sr(tmhd)@sub 2@. The deposition process was controlled by chemical reaction at < 500 @super o@C, and by mass transfer above 550 @super o@C. Decomposition of Ti(O-i-C@sub 3@H@sub 7@)@sub 4@ was enhanced with increasing rf power, but that of Sr(tmhd)@sub 2@ was greatly affected by the substrate

temperature. A deposition mechanism of SrTiO@sub 3@ was proposed based on the decomposition rates of Ti(O-i-C@sub 3@H@sub 7@)@sub 4@ and Sr(tmhd)@sub 2@.

**TF-MoP19 Investigation of Ti and Cu Atom Density in Magnetron Sputtering Process Using Atomic Absorption Spectroscopy**, ***E. Augustyniak***, *S.V. Filimonov, C. Lu,* Intelligent Sensor Technology

A hollow cathode lamp based atomic absorption monitor is applied to measure the relative atom density in a planar DC magnetron sputtering system for various Argon pressures (from 1 to 30 mTorr) and input powers (from 0.3 to 20 W/ cm@super 2@). Deposition rates for Ti and Cu are monitored with a piezoelectric microbalance in order to infer the absolute atom densities. Absorption versus deposition data obtained from thermal evaporation of the same materials at Argon pressures in the range from 10@super -6@ to 10@super -3@ Torr are used to estimate the degree of thermalization in sputtering process. Near the target, at distance shorter than one mean-free path of sputtered atoms, the film grows mostly due to deposition of energetic atoms. However, even in this region the thermalized atoms have a significant contribution to the total absorption of resonant radiation. The experimental data show that the reduction of local number density caused by the introduction of a nearby sink for sputtered atoms, e.g., a substrate or a sensor, can be as much as 50%. Ambient gas heating in the vicinity of the target at high input power results in suppressed thermalization and decreased sensitivity of atomic absorption. The combination of atomic absorption spectroscopic and gravimetric (by piezoelectric microbalance) measurements can be a powerful tool for sputtering process characterization.

**TF-MoP20 A Study of the Effect of Low Dielectric Constant PAE-2 by Plasma Treatment**, *T.C. Chang,* National Nano Device Laboratory, Republic of China*;* ***M.F. Chou****,* National Chiao Tung University, Republic of China*; T.W. Hsiao,* National Yun-Lin University, Republic of China*; J.Y. Lin,* National Yun-Lin University, Republic of China, Taiwan, Republic of China*; C.Y. Chang,* National Chiao Tung University, Republic of China, Taiwan, Republic of China*; M.S.K. Chen, A. Tuan,* Air Product Company, Republic of China, Taiwan, Republic of China*; S. Chou,* San Fu Company, Republic of China

As the device dimensions continue to shrink to 0.25um and below, the interconnect delay becomes a limiting device factor and increases device speed. Integrating a low dielectric constant ILD into device is a way to reduce the interconnect delay time constant. Low density material such as PAE-2 can offer lower dielectric constant than conventional silicon dioxide insulator. The PAE-2 film has the functional groups with oxygen linkage, so an oxidative post treatment could enhance the properties of PAE-2 film. In this work, we study the effect of post-plasma treatment of PAE-2 film characteristics. The dielectric constant of PAE-2 film is found to be reduced with the application of O@sub 2@ plasma treatment. Also, the optimized condition of O@sub 2@ plasma treatment is obtained. A model is proposed to explain the effect of post-plasma treatment in PAE-2 film.

**TF-MoP21 Porous Polycrystalline Silicon Micro-Electronic Sensor**, ***P.G. Han****,* The City University of Hong Kong, China*; H. Wong,* The City University of Hong Kong, China, Hong Kong, China*; M.C. Poon,* The Hong Kong University of Science & Technology, China, Hong Kong, China*; N. Wang,* The City University of Hong Kong, China, Hong Kong, China

Porous Polycrystalline Silicon(PPS)-based microelectronic sensors have been explored in our laboratory. Using n-type epitaxial silicon wafer as the substrate, undoped poly-Si films with thickness of 700 nm were deposited by thermal decomposition of silane gas(SiH@sub2@) in a low pressure chemical vapor deposition(LPCVD) reactor. Boron was then diffused into the samples at 950°C for 20 min to form a p-type polycrystalline silicon film. The poly-Si film was selectively anodized in HF(50%vol):C@sub2@H@sub5@OH(96%vol)=1:1 solution with Pt eletrode at a current density of 10mA/cm@super2@ for 10 min at room temperature.The sensitivity of the sensors based on this PPS has been characterized and annlyzed at different ambient pressures and temperature as well as gas species. Results show that the current increases remarkably as the pressure decreases and over two order of magnitude change has been detected when the vacuum pressure was pumped to 10@super -2@ ATM. We find that both ethanol and acetone vapors can make the current increase obviously. In acetone vapor, the device even behaves like a diode. The analysis of the electrical conduction mechanisms of the sensors in vaccum envirement and organic vapors will be conducted in this paper. In addition, since the fabrication process is simple, and the sensor characteristics are repeatable and reproducible, it can be easily integrated with the VLSI technology.

**TF-MoP22 Thickness and Index Measurement of Transparent Thin Films using Neural Network processed Reflectance Data**, ***M.F. Tabet****, W.A. McGahan,* Nanometrics Inc.

Artificial neural networks and the Levenberg-Marquardt algorithm are combined to calculate the thickness and refractive index of transparent thin films from spectroscopic reflectometry data. A neural network is a set of simple, highly interconnected processing elements imitating the activity of the brain which are capable of learning information presented to them. Reflectometry has been used by the semiconductor industry to measure thin film thickness for decades. Modeling the optical constants of a film in the visible region with a Cauchy dispersion model allows the determination of both thickness and refractive index of most transparent thin films from reflectance data. In this work Artificial neural networks are used to obtain good initial estimates for thickness and two Cauchy parameters An and Bn, these estimates are then used as the starting point for the Levenberg-Marquardt which does a few iterations to find the final solution. This measurement program was implemented on a Nanometrics NanoSpec 8000XSE and will measure thickness and index of transparent films in the range of 1000 to 16000 Å in an average of four seconds.

**TF-MoP23 Characterization of PECVD Hydrogenated Amorphous Silicon (a-Si:H)**, ***T.C. Ang****,* Nanyang Technological University, Singapore, Republic of Singapore*; M.S. Tse,* Nanyang Technological University, Singapore*; L.H. Chan,* Chartered Semiconductor Manufacturing Ltd., Singapore, Republic of Singapore*; J.L. Sudijono,* Chartered Semiconductor Manufacturing Ltd., Singapore

Unhydrogenated amorphous silicon has a high defect density and this prevents it from being useful for electronic devices. Hydrogen incorporation in a-Si eliminates defects and the quality of a-Si:H depends on the way the hydrogen is incorporated rather than its content in the film. The type of plasma used and the deposition conditions determine the nature of the hydrogen bonding and the film quality. In this paper, the film characteristics of PECVD a-Si:H deposited using pure silane plasma and silane plasma diluted with argon at different deposition temperatures are studied in terms of the deposition rate, hydrogen content, refractive index and film morphology. Fourier transform infrared spectroscopy (FTIR) spectra showed that in a-Si:H films deposited with argon dilution of the silane plasma, hydrogen is bonded to Si only in monohydride groups (SiH) whereas with a monosilane glow discharge plasma, the spectra revealed the presence of SiH, SiH@sub 2@ and (SiH@sub 2@ + SiH@sub n@) bonds. Higher hydrogen content was measured in low temperature deposited films. The RI and absorption coefficient (n, k) values of low temperature deposited films were lower and are consistent with the higher hydrogen content observed in the FTIR spectra. This is mainly due to the decreasing Si densities of the films due to the formation of Si-H and SiH@sub n@ (n>1) bonds in the films. Atomic force microscopy (AFM) was used to investigate the effect of deposition temperature on the film morphology and results showed that smaller grain sizes and tighter packing densities were characteristic of higher temperature deposited films. Results obtained through FTIR and spectroscopic ellipsometry (SE) show a correlation between the hydrogen content and the RI. Films deposited at higher deposition temperatures have lower H content and higher RI values as compared to lower temperature deposited films. Results from electrical tests on a-Si:H films deposited at different temperatures revealed changes in film quality which are consistent with the FTIR and SE observations. @FootnoteText@ W. Beyer and H. Wagner, J. non-crystall. Solids 59/60, 161 (1983). W. Beyer, Tetrahedrally-Bonded Amorphous Semiconductors, Ed. D. Adler and H. Fritzsche, Plenum Press, New York, 129, 1985. W. Beyer, Physica (Utrecht), 170B, 105,1991. R. A. Street, Hydrogenated Amorphous Silicon, Cambridge Solid State Science Series (Cambridge University Press, Cambridge, 1991). Akihisa Matsuda, Plasma Physics Control Fusion, 39, pp.A431,1997.

**TF-MoP24 The Analysis of Silicon Oxynitrides with Spectroscopic Ellipsometry and Auger Spectroscopy, Compared to Analyses by RBS, and FTIR**, ***H.G. Tompkins****, R. Gregory, P.W. Deal, S.M. Smith,* Motorola, Inc.

This work addresses the issue of whether spectroscopic ellipsometry, using the effective medium approximation (SE-EMA), may be used meaningfully to analyze PECVD silicon nitride films. We use RBS and FTIR as reference methods and compare the results to the results of SE-EMA analyses and Auger analyses. The results are that Auger analysis, using properly determined sensitivity factors, gives compositions which are within the uncertainty of the reference methods. SE-EMA, on the other hand, always overestimates the oxide contribution and underestimates the nitride contribution. Probable causes are discussed.

**TF-MoP25 Density Measurement of Thin Glass Layers for Gas Barrier Films**, ***N. Fukugami***, *H. Nishino, M. Yanaka, Y. Tsukahara,* Toppan Printing Co., Ltd., Japan

In the food packaging industry, there is an increasing demand for transparent gas barrier films consisting of a 10 ~ 100 nm thick glass layer deposited on a polymer substrate. An advantage of such films is that microwaves and light waves can penetrate. It was confirmed that our glass layer had no macro defects or pinholes, and the gas penetration mechanism was mainly due to a nano structure of the glass layer.@footnote 1@ It is anticipated that the density of the glass layer is an important factor to characterize such nanostructures. Therefore, an accurate density measurement of thin layers with thicknesses ranging from 10 nm to 100 nm was needed. We used a weight-volumetric method, and obtained the density with three digits accuracy. First, densities of SiOx layers which were deposited by vacuum evaporation on Si (100) surfaces were measured. The Si surfaces were used because they were well-characterized and smooth. The volume of a layer was obtained as a product of the layer thickness and its area. The area was defined by a window which was located in front of the Si substrate during the deposition. The thickness was obtained by measuring the edge height of stripes made over the entire surface by photolithography process. Dektak (Dektak3030, Nihon Shinku Gijutsu Inc.) was used for the edge height measurement. A weight of the layer was measured by Ultramicrobalance (Sartorius supermicro, Sartorius Inc.). The densities thus obtained were 2.15±0.01g/cm@super 3@ and 2.16± 0.03g/cm@super 3@ for layers with slightly different deposition conditions. Next, a novel method was invented for the density measurement of thin glass layers deposited on polymer substrates. The method made use of the fact that parallel multiple cracks were induced in the glass layer when the film was stretched.@footnote 2@ The thickness of the glass was obtained by measuring the depths of crack openings. The accuracy of the measurement was also investigated. @FootnoteText@ @footnote 1@B. Henry, et. al., presented at the 41st Society of Vacuum Coaters Technical Conference (April 1998). @footnote 2@M. Yanaka, Y. Tsukahara, N. Nakaso and N.Takeda, accepted to the J.Mat.Sci., 1998.

**TF-MoP26 Accurate and Rapid Determination of Thickness, n and k Spectra, and Resistivity of ITO Films**, *K. Zhang,* EG&G, US; ***R. Forouhi***, *I. Bloomer,* n&k Technology, US

The transparent conductor, indium tin oxide (ITO), is an important thin film component of flat panel displays. An optimum ITO film should be both highly transparent to visible wavelengths and at the same time, conductive. In practice, however, a trade-off exists between these two attributes, making it difficult to produce a film that simultaneously meets both demands. In order to achieve the optimum balance between these properties, an effective method of characterizing ITO films is necessary. In this talk we will present results of a new measurement technique that simultaneously determines, thickness, the spectra of the refractive index (n) and extinction coefficient (k) from 190 to 1100 nm, and the energy band gap, of ITO films deposited on either transparent or opaque substrates. In addition, we will demonstrate how the film's resistivity can be correlated to the film's extinction coefficient. This technique is based on wide-band spectrophotometry, combined with spectral analysis incorporating the Forouhi-Bloomer dispersion equations for n and k.@footnote 1,2@ The measurement technique is non-destructive and takes only a few seconds. @FootnoteText@ @footnote 1@A.R. Forouhi and I. Bloomer, Phys. Rev. B, 34, 7018 (1986). @footnote 2@A.R. Forouhi and I. Bloomer, Phys. Rev B, 38, 1865 (1988).

**TF-MoP27 Mechanical Properties and Strengthening Mechanisms of Pure Iron Implanted with Metal Ions**, ***D. Yang***, University of Alabama; *Q. Xue,* Lanzhou Institute of Chemical Physics, China

Ion implantation can alternates surface composition and microstructures of metals dramatically, consequent change of physical and chemical properties of ion implanted layers appears as a result of it. The first noticeable effect of implantation is variation of surface residual stress and microhardness. To further understand the effect of metal ion implantation on mechanical properties and strengthening mechanisms, Cr, Mo, W, Ni, Al ion implantation into pure iron was performed on a Metal Vapor Vacuum Arc (MEVVA) source implanter at doses of 5×10@super 16@ ions/cm@super 2@, 1×10@super 17@ ions/cm@super 2@ and 3×10@super 17@ ions/cm@super 2@. The atomic concentration, element distribution, phase structure, surface residual stress and microhardness were measured by Auger Electron Spectroscopy (AES), X-ray Diffractometry and microhardness tester respectively. The relationship among the atomic radius, residual stress and hardness was studied and discussed. It was shown that the highest atomic concentrations in the implanted layers were between 6 at.% to 38 at.% depending on the elements implanted and implantation doses. The higher and bigger the implantation doses and differences in radius between pure iron and implanted element atoms are, the larger the highest atomic concentration in the implanted layers. In addition, the ion implantation resulted in residual compressive stresses of 663-957 MPa and an increase of 5%-43% in microhardness of pure iron surface. Ion implantation dose was not the only factor influencing surface stress and hardness. The decisive factor was the existing format of ion implanted element, i. e. if the implanted element existed as solid solute the residual compressive stress would be higher and the hardness would be lower; if it existed mainly as compounds then the stresses would be lower and the hardness higher.

**TF-MoP28 The Dislocation Network Developed Deep in Titanium Nitride by Ion Implantation**, ***A.J. Perry***, A.I.M.S. Marketing, Japan; *D.E. Geist,* Analytical Reference Materials International; *Y.P. Sharkeev,* Russian Academy of Sciences, Russia; *S.V. Fortuna,* Tomsk State University of Architecture & Building, Russia

After treatment by ion implantation, the implanted ions in metallic materials reside in an implanted zone, IZ, extending to a depth of some hundreds of nanometers. Earlier work has shown that the momentum carried by the ions affects the material to far greater depths with a dislocation network extending many microns deep. Dense, thick coatings of titanium nitride can be deposited with low residual stress and low dislocation densities at high temperatures, some 950 C, without the application of substrate bias. Normally used for wear resistance, these coatings are an ideal model system for studying the nature of the IZ and the development of the dislocation network well below it. Studies have been carried out by TEM and glancing incidence XRD. The TEM study shows that implantation leads to the formation of sub-grains within the original grain structure without any grain comminution occurring. A mechanism is then proposed to explain the development of the dense dislocation network below the IZ which is studied by XRD as a function of ion species, acceleration voltage and dose in an extended series of samples. It is found that while any ion bombardment produces a dislocation network, there is a momentum threshold level before a compressive residual stress is developed. This stress is proportional to the ion momentum, reaching values as high as 3-4 GPa, i.e. as high as that found in materials made by PVD methods. Finally we find that there is a significant change in the stress in the substrate below the titanium nitride coating which, surprisingly, can be tensile or compressive and is sensitive to the ion species implanted.

## Thin Films Division
## Room 310 - Session TF-TuM

### Thin Films for Sensing and Data Storage

**Moderator:** S. Semancik, National Institute of Standards and Technology

8:20am **TF-TuM1 Thin Film Media Requirements in Ultrahigh Density Magnetic Recording**, ***D. Weller***, *A. Moser,* IBM Almaden Research Center **INVITED**

Rapidly increasing areal densities and data rates in hard disk magnetic recording impose stringent requirements on media and head materials. An imminent problem is media instability, which results from superparamagnetic effects. These effects become measurable in thin film granular magnetic recording media in the thickness range of about 10 nm and below. They lead to sizeable magnetic viscosity (logarithmic magnetization decay as function of measurement time) and to a write pulse width dependent coercivity. Examples of the latter measurements over 10 magnitudes in time (5 ns to 50 s) will be presented for typical CoPtCr magnetic recording media of variable thickness and at variable temperatures. The results will be discussed in the context of Neel-Arrhennius based decay models and recent micromagnetic modeling calculations. The thermal stability problem has led to a surge in interest in novel materials and media schemes. These include conventional but higher anisotropy media, perpendicular media, self assembled particle arrays and patterned media. We will discuss these various approaches in the context of ultimately achievable areal densities.

9:00am **TF-TuM3 Molecular Dynamics Analysis of Energy Modulated Deposition of Model GMR Materials**, ***H.N.G. Wadley***, *X.W. Zhou,* University of Virginia

Vapor deposited multilayers consisting of low electrical resistivity conductors sandwiched between ferromagnetic metals exhibit giant magnetoresistance (GMR). The best GMR properties are obtained from materials with flat interfaces and low intermixing between adjacent layers. Interfacial roughness and intermixing are sensitive to the deposition method and process conditions. A three dimensional molecular dynamics model has been developed and used to establish the relationship between the multilayer nanostructure and vapor deposition conditions, including incident atom angle, incident atom energy and substrate rotation. The results indicate that at low incident energies (e.g., 1 eV or below), an increase in the incident angle leads to a significant increase in the interfacial roughness (and even to void formation) due to a shadowing effect. The development of interfacial roughness was also found to be accompanied by an increase in intermixing. The high interfacial roughness formed during oblique, low energy deposition can be significantly reduced by substrate rotation. High incident atom energies were found to result in a lower interfacial roughness, but at the expense of increased intermixing caused by an atomic exchange mechanism. Under normal incidence conditions, an intermediate incident energy of between 1 and 2 eV resulted both in a low interfacial roughness and intermixing. The simulation methodology was used to explore the benefits of modulated incident energy deposition strategies. When thermal energy adatoms were used to deposit the first few monolayers of each new metal layer, intermixing by the exchange mechanism during subsequent hyperthermal energy deposition could be eliminated, and films with almost no interfacial roughness or intermixing could be grown over a wide incident angle range.

9:20am **TF-TuM4 Obtaining Optical Constants of Thin Ge@sub x@Sb@sub y@Te@sub z@ Films from Measurements of Reflectance and Transmittance**, ***D.V. Tsu***, Energy Conversion Devices

Chalcogenide thin films are currently used in Phase Change Rewritable Optical Recording media such as CD-RW and PD. These media have storage capacities 650 Mbytes, and can be rewritten over 1000 and over 100,000 times respectively. The second generation of Phase Change Rewritable media, using the DVD format, will extend that capacity to 4.7 GBytes/side. At the heart of this technology is the Chalcogenide layer, which undergoes a reversible change between amorphous and crystalline structures upon absorption of appropriate laser energy. The accompanying change in the optical constants [n,k] of the chalcogenide layer results in large reflectivity differences of the multi-layer media. Design of Phase Change optical media requires precise determination of [n,k] for both structures of the alloy over a wide spectral range. We have accomplished this task from measurements of [R,T] using the [air/film/substrate/air] configuration for films < 50 nm thick. The film's [n,k] are obtained by direct numerical inversion of the appropriate [a/f/s/a] expressions relating [n,k] to the measured [R,T]. Since R and T are highly non-linear in [n,k], calculating [n,k] provides multiple solutions at each wave length including the physical solution and also other mathematically relevant solutions. The other solutions can be used to determine the film's average thickness to an accuracy of ~ 1% or better. Using this technique we show that when as-deposited amorphous Chalcogenide films crystallize upon annealing they typically undergo a reduction in thickness of ~ 10%. We have developed software which can calculate [n,k] at 1800 wavelength points in less than 30 seconds. Attaining the correct film thickness requires only a few iterations. The calculation methodology does not include any assumption regarding the dispersion relation of the unknown film. This is critically important when multi-layered media structures are developed using new alloys.

9:40am **TF-TuM5 Process Monitoring of Hemispherical-Grained Polysilicon Thin Films for DRAM Applications**, *C. Hayzelden,* ***A. Bivas***, *C.L. Ygartua, K.C. Chan,* KLA-Tencor Corporation

The fabrication of hemispherical-grained (HSG) polycrystalline silicon was developed to increase the surface area (and storage capacitance) of Dynamic Random Access Memory (DRAM) devices. The increase in surface area (typically 2.4 x) is extremely sensitive to processing conditions. Therefore, to obtain high yields, process monitoring is of great importance. The use of spectroreflectometry and spectroscopic ellipsometry, described in this paper, provides a non-destructive method to monitor film fabrication. Using these two measurement technologies, the intensity and polarization state of the light reflected from the wafer surface is measured and analyzed over the wavelength range 220 to 800 nm. In the measurement analysis, a polysilicon layer is frequently modeled as a mixture of several materials, using the Effective Medium Approximation (EMA). Using the EMA model, with a composition of crystalline silicon, amorphous silicon, and a percentage of "voids", the analysis of measurement data yields values of the optical parameters (refractive index, extinction coefficient) and effective thickness of the HSG layer. In this paper, we present capacitance data from a series of HSG-processed wafers and report the observed correlation with the measured variables: percentage of "voids", effective thickness and optical parameters. The most appropriate variables to report for accurate process control will be discussed.

10:00am **TF-TuM6 Atomistic Scale Modeling of Ion Assisted Deposition of GMR Multilayers**, ***X.W. Zhou***, *H.N.G. Wadley,* University of Virginia

Giant magnetoresistance (GMR) is found in many ferromagnetic metal multilayers separated by thin copper films. Low interfacial roughness and intermixing are critical for obtaining the high giant magnetoresistance ratio needed for magnetic random access memories. A three dimensional molecular dynamics physical vapor deposition model has been developed and used to explore the effects of Ar- and Xe- ion assisted deposition of model Ni/Cu/Ni multilayers. The results indicate that the interfaces can be significantly flattened by ion assisted deposition provided the ratio of ion to metal atoms exceeds about 2.0. However, we found that as the assisting ion energy is increased, interfacial roughness decreases, but intermixing of the layers increases. A modulated ion energy / flux strategy for ion assisted deposition has then been investigated in which the ion beam energy / current was reduced while depositing the first few monolayers of each new metal layer. This strategy was found to successfully reduce both interfacial roughness and intermixing. The optimal ion to metal atom ratio and ion incident energy were identified.

10:20am **TF-TuM7 Thin Film Gas Sensors Based on Metal Oxides**, ***H. Meixner***, *M. Fleischer,* Siemens AG, Germany **INVITED**

Recently, research into the characteristics of semiconductor metal oxides that are stable at high temperatures with a view to providing reproducible detection of oxygen and reducing gases has intensified. First of all, there is a discussion of the specifics relating to these materials, for example the reduction of the effects of grain boundary barriers on the conduction mechanism, the reduction of the humidity cross sensitivities and also on the various reaction mechanisms. Then, the technology for constructing gas sensors of this kind will be described. Examples that have already been implemented, say, @gamma@ detection, O@sub 2@ measurements in the exhaust gases from incinerators, methane alarms, and air quality control as well as certain trends in development are discussed. Keywords: Metal Oxides, Thin Films, High Temperatures, Conduction Mechanisms, Gas Sensors

11:00am **TF-TuM9 A Study of Thermally-Defined Gas Sensing Films on Micromachined Arrays**, ***R.W. Walton***, *R.E. Cavicchi, J.D. Allen, S. Semancik,* National Institute of Standards and Technology

Film microstructure and interfacial composition of sensing materials can have a profound effect on the performance of solid state gas sensors. We report on an efficient, array-based study of property/performance relationships for conductometric gas sensing films done on micromachined devices called microhotplates. Four-element arrays were used primarily in this study. Two types of lithographic processes were employed to deposit the catalytic metal/semiconducting oxide sensing films; each method utilizes the localized heating and temperature control available for the individual elements of the microhotplate array. A CVD process uses the heater to activate surface reactions for selected-area film deposition. We also introduce a new lithographic process that involves coating the entire array with nitrocellulose and exposing chosen elements by eliminating their coatings with a high temperature pulse. Following deposition (by evaporation, sputtering), lift-off of the nitrocellulose removes unwanted material. In situ monitoring of both deposition processes makes use of built-in electrical contacts for measuring conductance. We demonstrate the power of this array approach for studying film deposition, thermal treatments (sintering), and sensor testing. CVD films of SnO@sub 2@ films were prepared by heating each 50µmx50µm hotplate to 500° C in a flow of tetramethyltin and O@sub @ in Ar. We correlate the effects of catalyst thickness (0-200 Å), annealing (to 700° C), and composition (evaporated Pd and Pt) with gas sensing response from 100° C to 500° C. SEM images show anneal treatments as low as 500° C change the morphology of the catalysts on the oxide grains to produce islands or porous structures that enhance sensitivity to reducing gases.

11:20am **TF-TuM10 Germanium Films and Their Application in Cryogenic Temperature Sensors**, *N.S. Boltovets,* State Research Institute "Orion", Ukraine*; J.P. McFarland, G.G. Ihas,* University of Florida*; R.V. Konakova,* ***V.F. Mitin***, Microsensor Co. Ltd., Ukraine

The principal objectives of our work were: (i) to prepare Ge films on GaAs suitable to develop temperature sensors; (ii) to study the structure and electrical properties of Ge films; (iii) to develop and design miniature wide-range thermometers capable of operating in the 30 mK to 300 K temperature range; and (iv) to investigate sensors at low temperatures and in high magnetic fields. The films have been prepared by Ge thermal evaporation in a vacuum on semi-insulating GaAs substrates. The electrical properties of the films obtained depended on the details of their fabrication. To fabricate a resistance temperature sensor capable of operating over a wide temperature range, several different conduction mechanisms are to be realized in the Ge film, each of which providing thermo-sensitivity in its own temperature range. This may be achieved through multi-component doping and compensation of the Ge film. At least three main sources of extra charge carriers exist in the Ge films on the GaAs substrates; namely, Ga and As atoms and structural defects. A relationship between them is determined by the film growth conditions. By varying the conditions during the film fabrication, one can prepare Ge films having different doping levels and degrees of compensation, i.e., different thermo-sensitivities. In this way, temperature sensors having different thermometric characteristics and capable of operating over different temperature ranges can be produced. Films with a monotonic temperature dependent resistance in the 30 mK to 300 K range have been fabricated. We present the miniature temperature sensor design based on Ge films, and results of sensor investigation in this temperature range and in magnetic field up to 6 Tesla. We also discuss the mechanism of conductivity in the films responsible for magneto- and thermo-sensitivity.

11:40am **TF-TuM11 Characterization of Sol-Gel Prepared WO@sub 3@ Thin Films as a Gas Sensor**, ***M.Z. Atashbar***, *Y. Li, M.K. Ghantasala, W. Wlodarski,* RMIT University, Australia

Increasing awareness of well human being to environmental issues has attracted many scientific researches in the field of gas sensing. Tungsten trioxide (WO@sub 3@) thin films have promising electrical and optical properties for different applications, such as electrochromic displays, photolysises and gas sensing. The change of its conductivity due to the gas exposure has made itself a excellent candidate for nitrogen oxide (NO@sub x@) and ozone (O@sub 3@) sensing applications. In this study, the effect of the structure, micro-morphologies and composition of the WO@sub 3@ film on NO@sub x@ and O@sub 3@ sensing properties has been investigated. WO@sub 3@ thin films were prepared from starting precursors via sol-gel route in dry nitrogen atmosphere (@<=@10%RH). The used precursors was tungsten ethoxide (W(OC@sub 2@H@sub 5@)@sub 3@) with an analytic purity, which was dissolved in different analytical solvents. After ultrasonic mixing at room temperature for 1 hour the precursor solution was dropped on substrates and spin-coated at different speeds. The film thickness (20 to 200 nm) was controlled by changing the solution concentrations and spinning speed. After annealing at temperature of 400°C to 700°C crack-free WO@sub 3@ thin films were obtained. The morphology, microstructure, crystalline structure and composition of the deposited films were analyzed by SEM, XRD and RBS. The SEM analysis showed that that film is porous and the grain size is the rang of 20nm to 100nm. The RBS results indicated that the film is sub-stoichiometric. XRD analysis showed that the films are polycrystalline in nature. A detailed analysis of these results on the O@sub 3@ and NO@sub x@ sensitivity of the film has been discussed. The electrical resistance of the film has been examined for the detection of ozone in ppb level and NO@sub x@ ppm level.

## Vacuum Metallurgy Division
## Room 328 - Session VM+TF-TuM

### Advances in Hard and Superhard Coatings

**Moderator:** A. Inspektor, Kennemetal, Inc.

8:20am **VM+TF-TuM1 Deposition, Structure, and Properties of Superlattice Thin Films**, ***S.A. Barnett***, *A. Madan, P. Yashar, I. Kim,* Northwestern University **INVITED**

In this talk, superlattice thin films with nitride/nitride, metal/nitride, and oxide/oxide layers are described. Processing issues for high-rate superlattice deposition using reactive magnetron sputtering are described, including reactive-gas partial pressure control for obtaining stoichiometric layers and use of substrate bias to achieve ion bombardment densification. The stability of the layered structures at elevated temperatures is described; it is found that nanometer thick layers can exhibit excellent stability in cases where the relevant phase diagram shows little miscibility. The key materials criteria for obtaining hardness enhancements are delineated by making comparisons between different superlattice systems. Hardness predictions based on dislocation glide mechanisms are discussed. For cases where both superlattice layers have the same structure, a substantial difference between the layer shear moduli is required to limit dislocation motion and thereby strengthen the material. Superlattices where the layers have different structures, such that there is no common dislocation glide system, can also exhibit large hardness enhancements.

9:00am **VM+TF-TuM3 In-situ and Ex-situ Ellipsometric Analysis of Cr, CrN, Cr@sub 2@N Thin Films**, *D.M. Mihut, S.R. Kirkpatrick,* ***S.L. Rohde***, University of Nebraska, Lincoln

Chromium nitride thin films have technological applications in the tool and decorative coating industries, as well as providing an "environmentally-friendly" alternative to hard chrome coatings due to their unique combination of properties such as: low cost, high hardness (1600 - 3000 HK), excellent wear, corrosion, and oxidation resistance (up to 800 °C). An array of chromium and chromium nitride film monolithic and multilayered films were deposited in a ultra-high vacuum chamber equipped with an unbalanced magnetron sputtering system that combines the advantages of high-rate magnetron sputtering with high-flux, low energy ion bombardment. Ellipsometric analysis of the films was carried out by modeling layers of both stoichiometric and off-stoichiometry Cr, CrN and Cr@sub2@N thin films deposited on silicon. The ex-situ ellipsometry measurements were compared with X-ray diffraction measurements, and in-situ obtained ellipsometric information. The optical constants for CrN and Cr@sub2@N obtained using optical ellipsometry are given and compared with the optical constants for CrN and Cr@sub2@N found in the literature, and the potential of using ellipsometry in the monitoring and/or control of ionized PVD processes explored.

9:20am **VM+TF-TuM4 AlN/cBN Magnetron Sputtering: Effects on Adhesion and Phase Stabilization**, ***W. Otaño***, *L.J. Pilione, R. Messier,* Pennsylvania State University*; J.J. Santiago-Avilés,* University of Pennsylvania*; G. Lamaze,* National Institute of Science and Technology

The deposition of cubic boron nitride (cBN) thin films is of interest from a technological and fundamental point of view. It has been well established that the cubic phase stabilization depends on the energetic bombardment of the growing film. As a result of this bombardment the films show high stress levels that eventually produce delamination from the substrate. It is therefore interesting to consider atomic additions and compliant interfaces as alternative pathways to reduce the cBN stress level and/or improve film adhesion. With this purpose cBN thin films were co-deposited with Al

reactively sputtered. The effects of the addition of aluminum and/or the use of AlN interlayers in the stabilization and adhesion of cBN films will be presented. The BN films were deposited by rf unbalanced magnetron sputtering and the substrate was biased using a low frequency dc pulsed excitation signal. Films with over 70% of the cubic phase, as measured by FTIR, were deposited at low negative bias voltages. A second dc pulsed power supply was used to reactively sputter the aluminum. AlN was added as an interlayer between the substrate and the BN film as well as co-deposited at different sputtering powers. The films were analyzed by FTIR, RBS and neutron depth profiling. It was found that the addition of Al to the BN films leads to a destabilization of the cubic phase for AlxB1-xN compositions above x=0.04. AlN interlayers deposited at specific pressures were found to prevent the delamination of the cBN films. A 0.7 micron multilayer coating of AlN/cBN was prepared that did not delaminate from the substrate.

9:40am **VM+TF-TuM5 Energetics of Cubic Boron Nitride Deposition**, ***R. Clarke***, *D. Litvinov,* University of Michigan **INVITED**

As a structural analog of diamond, cubic boron nitride (c-BN)is attracting increasing interest as an ultrahard coating material. An ongoing challenge towards exploiting the favorable properties of c-BN, including its chemical intertness and high thermal conductivity, is its tendency to build up substantial levels of stress at practically useful thicknesses of a few microns. If this stress is not remediated, it can lead to loss of adhesion. In this talk we present recent results demonstrating our approach to characterizing and controlling the intrinsic stress in c-BN coatings. Through an improved understanding of the kinetics of c-BN growth, using dc-biased ECR-assisted sputtering, we have achieved highly adhesive coatings on Silicon with film thicknesses up to 2µm. A novel multibeam optical wafer curvature method allows us to track, in-situ, the stress build-up during growth, and to implement 'reduced-bias' conditions after the initial nucleation and coalescence of c-BN islands. Reducing the kinetic energy of arriving nitrogen ions in this way (to ~ 50eV), leads to fewer defects in the film, reduced levels of stress, and higher growth rates, compared to values obtained at bias conditions necessary to initiate c-BN growth. Work partly supported by ONR grant N00014-94-J-0763, and by k-Space Associates Inc.

10:20am **VM+TF-TuM7 Deposition and Characterization of Ultra Thin CNx Films as a Thin-Film Disk Overcoat**, ***X. Chu***, *Z.D. Yang, J.F. Ying, S. Wang, B. Zhang,* MMC Technology Inc.

The deposition of CNx films has received great attention recently because of the potential of this material to have mechanical properties similar to diamond. One practical application of magnetron sputtered CNx films is for use as a protective coating for thin film magnetic recording disks. Ever increasing magnetic recording density requires not only a robust head-media interface, but also minimum spacing loss due to fly height, carbon thickness and magnetic layer thickness. A functional overcoat with a thickness of 50 to 100 Å is needed for the next generation recording medium. In this paper, we investigate sputtering process parameter effects on CNx film structure and mechanical properties. Target power, N% in the sputter gas, substrate temperature, and substrate bias were varied and correlated to film properties. XPS and Raman spectrum were used to study the bonding structure of the film. Sputtered CHx and CHNx films with 80 Å thicknesses and ion beam deposited CHx films were also studied for comparison. Nano-scratch wear tests showed that the 80Å film had the best wear property with 10-15% N in the gas. CNx films appear to be more wear resistant than CHx and CHNx films based on the nano-scratch test. Tribology properties of lubricated disks were tested using Contact Start Stop (CSS) testers and CNx carbon wear results can be correlated to the AFM nano-wear test. CHx films also showed good CSS results, suggesting that lubricant - carbon interaction is another important factor in head - media tribology.

10:40am **VM+TF-TuM8 Carbon and Carbon Nitride Films Prepared by Low-Energy, Isotopically-Mass-Separated, Negative C@sub 2@@super -@ and CN@super -@ Ions**, ***N.T. Tsubouchi***, *A.C. Chayahara, A.K. Kinomura, C.H. Heck, Y.H. Horino,* Osaka National Research Institute, AIST, Japan

Amorphous carbon (a-C) and carbon nitride (a-CN@sub x@) films were prepared by ion beam deposition using isotopically mass-separated, hyperthermal (50-400 eV) negative ion species such as @super 12@C@sub 2@@super -@ and @super 12@C@super 14@N@super -@ under ultra high vacuum (UHV) condition. Variation of optical constants as a function of ion's kinetic energy was investigated in the infrared-visible light region (0.8-1.5 eV). Optical band gaps of the films were estimated from optical constants. For the amorphous carbon films, the gaps were about 1.0-2.3 eV depending on kinetic energy of negative carbon ions. For the CN films, the values which did not almost depend on kinetic energy were about 0.8 eV.

11:00am **VM+TF-TuM9 Investigation on Multilayered Chemical Vapor Deposited Ti/TiN Films**, ***J.C. Hu,*** National Tsing Hua Univ., Rep. of China, Republic of China; *T.C. Chang,* National Nano Device Lab, Rep. of China, Republic of China; *L.-J. Chen,* National Tsing Hua Univ., Rep. of China, Republic of China; *Y.L. Yang,* National Nano Device Lab, Rep. of China, Republic of China; *P.T. Liu,* National Chiao Tung Univ., Rep. of China, Republic of China; *S.Y. Chen,* National Tsing Hua Univ., Rep. of China, Republic of China; *C.Y. Chang,* National Chiao Tung Univ., Rep. of China, Taiwan, Republic of China

As the device dimensions scale down to deep submicron level, chemical vapor deposition (CVD) for TiN films provided excellent step coverage and uniformity. Cu is likely to replace Al for interconnect metallization in future integrated circuits. On the other hand, the CVD-TiN films are usually of columnar structure. As a result, the fast diffusion of Al (or Cu) and Si atoms along TiN grain boundaries would degrade the device performance severely. In the present study, a novel multilayered CVD-Ti/TiN structure is formed to alleviate the grain boundary effects. To investigation the barrier property of the multilayered Ti/TiN films, junction leakage current was also measured. All the films were deposited by CVD processed in a MRC multichamber cluster tool, using TiCl@sub 4@, NH@sub 3@ and H@sub 2@ as reactants. The Ti and TiN films were deposited by plasma enhanced CVD and low pressure CVD, respectively. In order to reduce chlorine concentration of the films, NH@sub 3@ plasma post-treatment was applied to multilayered CVD-Ti/TiN films. In addition, electroless deposition of Cu was deposited on the multilayered CVD-Ti/TiN films. Transmission electron microscopy and X-ray diffractometry were utilized to investigate the microstructure and crystal orientation. Auger electron spectrocopy was applied to determine the stoichiometry and uniformity along the depth direction. The morphology was studied by a field emission scanning electron microscopy. Electrical measurement was used by HP-4145. The enhanced multilayered Ti/TiN stack found to be a robust barrier against Al/Si interdiffusion. It also improved the electrical property of the films. The resistivity of the film was found to reduce from 240 to 120 µm@OMEGA@-cm by multilayered Ti/TiN structure with the NH@sub 3@ plasma post-treatment. The leakage current can also be kept low enough for device application. In addition, the thermal stability of electroless Cu/mutilayered (CVD-Ti/TiN)/TiSi@sub 2@/Si structure was improved.

11:20am **VM+TF-TuM10 Chemical Vapor Deposition of Metal (Ti) and Ceramic (TiO@sub 2@, TiN) Thin Films via Gas-Phase Reaction of Titanium Tetrachloride and Sodium Metal Vapor**, ***J.H. Hendricks***, *M.I. Aquino, J.E. Maslar, M.R. Zachariah,* National Institute of Standards and Technology

A new route for Chemical Vapor Deposition (CVD) of metal and ceramic thin films has been demonstrated. This novel method involves the use of a low pressure coflow diffusion reactor to react sodium vapor with titanium tetrachloride in the presence of a non-reactive gas (Ar) or a reactive gas (N@sub 2@, O@sub 2@). This reaction chemistry is described by the following general equation: (mn)Na + nMX@sub n@ --@super Ar@--> (M)@sub n@ + (nm)NaX. Here, Na is an alkali metal (e.g. Na, K, Cs, or Rb), M is a metal (e.g. Ti, Ta, Pt, W, ...) or non-metal (e.g. B, C, Si, ...), X is a halogen (e.g. F, Cl, Br, or I), Ar is a non-reactive gas (e.g. Ar or He) and m and n are integers. In this reaction, the alkali metal strips halogen from the metal or non-metal halide. The metal or non-metal is then free to form a thin film on a substrate placed in the reaction zone. This chemistry should be generic for the deposition of a wide class of metallic and ceramic thin films, and it is suggested that this technique could be used to grow superhard BN and CN thin films at temperatures which are significantly lower than conventional CVD techniques. Guided by theoretical modeling, reactant concentrations and substrate temperatures were adjusted to prevent salt (NaCl) incorporation into the deposited thin films. Using the described techniques, we have now produced Ti and TiN thin films on Cu substrates at 610 °C, and TiO@sub 2@ thin films on Si substrates at 600 °C. These temperatures are considerably lower than the (1000 to 1200) °C required for conventional CVD of Ti (by decomposition of titanium tetraiodide). The quality and composition of the thin films were analyzed by scanning electron microscopy (SEM), energy dispersive x-ray spectrometry (EDS), x-ray diffraction (XRD), Raman spectroscopy, transmission electron spectrometry (TEM), and selected area electron diffraction (SAED). Future work will focus on the use of this novel technique to grow CN and BN thin films.

11:40am **VM+TF-TuM11 Low Energy Ion Beam Deposition of Oriented Diamond Microcrystallites**, ***P.K. Tse***, *R.W.M. Kwok, K.M. Lui, W.M. Lau,* The Chinese University of Hong Kong, China

Ion beam deposition provides an additional control of film properties over the chemical vapor deposition (CVD) via the change of ion beam energy. In this study, low energy ion beam deposition of carbon films on silicon in the ion energy range of 200 - 1050 eV was studied. The ion beam was characterized by a Faraday cup equipped with a retarding lens. The films were characterized using X-ray photoelectron spectroscopy, characteristic electron energy loss analysis, and atomic force microscopy. It was found that graphitic films, amorphous carbon films and oriented diamond microcrystallites could be obtained separately at different ion beam energies. Highly oriented diamond microcrystallites were deposited on Si (100) wafer at energy of 200eV and substrate temperature of 420°C. The ion beam deposition will be used as a diamond seeding process which will be followed by a typical hot filament CVD process, for the growth of oriented diamond films on Si (100).

## Thin Films Division
## Room 310 - Session TF-TuA

### In-situ Characterization of Thin Films

**Moderator:** J.J. Nainaparampil, Air Force Research Laboratory

2:00pm **TF-TuA1 Real Time Ellipsometry Study the Deposition of Barium Strontium Titanate Thin Films**, ***Y. Gao***, *A. Mueller, E.A. Irene,* University of North Carolina, Chapel Hill*; O. Auciello,* Argonne National Laboratory, U. S. A.*; A.R. Krauss,* Argonne National Laboratory

(Br, Sr)TiO@sub 3@ (BST) has been considered to be a candidate high K dielectric material in dynamic random access memory(DRAM) capacitors.@footnote 1@ However, the interface layer formed between the BST thin film and substrate is an obstacle to obtain the desired dielectric constant and/or leakage current level for high density DRAM applications. Therefore, a clear understanding and then control of the interface reaction between a high K dielectric film and the substrate is tantamount for further progress in high K film technology. In previous work@footnote 2@ in our laboratory, the oxygen extraction and interface reactions of YBCO superconductor thin films on different substrates have been studied using real time ellipsometry. The results of this study comprise information on film growth kinetics and the oxygen in- and out-diffusion mechanism. Presently a similar study is ongoing on the interface reaction in ion sputtered BST thin films on Si substrates. The results of this study to be presented include: 1. real time film growth kinetics 2. sensitivity of bulk film composition to oxygen background pressure 3. interface reactions with Si and with various buffer layers 4. static and high frequency dielectric constants as well as leakage currents for BST films from various growth conditions 5. optical properties, composition, structure and morphology of BST films from various growth and post-deposition annealing conditions. @FootnoteText@ @footnote 1@D.E. Kotecki, Semiconductor International, Nov. 1996, pp109-116; @footnote 2@A. Michaelis and E.A. Irene et. al, Journal of Applied Physics, June 15, (1998).

2:20pm **TF-TuA2 Optical Constants of Crystalline WO@sub 3@ Deposited by Magnetron Sputtering**, ***M.J. DeVries***, *C. Trimble, T.E. Tiwald, D.W. Thompson, J.A. Woollam,* University of Nebraska, Lincoln*; J.S. Hale,* J. A. Woollam Co., Inc.

Crystalline WO@sub 3-x@ is an infrared (IR) electrochromic material with possible applications in satellite thermal control and IR switches. Optical constants of electrochromic materials change upon ion intercalation, usually with H@super +@ or Li@super +@. Of primary concern for device design are the optical constants in both the intercalated and unintercalated states. In-situ and ex-situ ellipsometric data are used to characterize both the deposition process and the optical constants of the film. Ex-situ data from a UV-Vis-NIR ellipsometer are combined with that from a mid-infrared FTIR-based ellipsometer to provide optical constants over a spectral range of 190 nm to 30 µm.

3:00pm **TF-TuA4 Ordered Binary Oxide Films: V@sub 2@O@sub 3@ (0001)/Al@sub 2@O@sub 3@/Mo(110)**, ***Q. Guo***, *D.Y. Kim, S.C. Street, D.W. Goodman,* Texas A&M University

Ordered binary oxide films, vanadium oxide on aluminum oxide, on the (110) molybdenum surface have been prepared in ultra-high vacuum conditions and characterized by various surface analytical techniques. Results from Auger electron spectroscopy, low energy electron diffraction, high-resolution electron loss spectroscopy, X-ray photoelectron spectroscopy and ion scattering spectroscopy indicated that the vanadia films grew on the Al@sub 2@O@sub 3@/Mo(110) surface epitaxially as V@sub 2@O@sub 3@(0001). Results from electronic structure measurements show an increase in energy of the a@sub 1g@ level in the 3d band at 100 K, which is one possible contributor to the metal-insulator transition in V@sub 2@O@sub 3@.

3:20pm **TF-TuA5 In-Situ Thin Film Characterization**, ***J.M. Gibson***, University of Illinois, Urbana **INVITED**

Control and understanding provide the two primary motivations for in-situ film characterization. For vacuum deposition, one powerful in-situ probe is the high-energy electron beam. Reflection High-Energy Electron Diffraction(RHEED) is a well-known method, used in Molecular Beam Epitaxy(MBE), for both purposes. Direct imaging using high energy electron beams forms the basis of transmission electron microscopy (TEM), which provides far more microstructural information than RHEED. Due to the instrumental complexity and invasiveness of the technique, however, in-situ TEM is directed at understanding, rather than controlling growth. I illustrate with examples from my group's work which includes: understanding the growth of very thin oxides, both on metals@footnote 1@ and semiconductors;@footnote 2@ the nature of small particle sintering in the formation of nanophase metallic thin films;@footnote 3@ island growth and stress@footnote 4@ in epitaxial Ge on Si; and AlN epitaxy on sapphire.@footnote 5@ The emphasis is that understanding through in-situ microscopy can lead to control, without in-situ probes, through better understanding of growth processes. @FootnoteText@ @footnote 1@J. C. Yang and J. M. Gibson, Appl. Phys. Lett. 70, 3522 (1997). @footnote 2@X. Chen and J. M. Gibson, Appl. Phys. Lett. 70, 1462 (1997). @footnote 3@M. Yeadon, J. C. Yang, M. Ghaly, D. Olynick, R. Averback, and J. M. Gibson, Appl. Phys. Lett. 71, 1631 (1997). @footnote 4@R. D. Twesten and J. M. Gibson, Phys. Rev. B 50, 17628 (1994). @footnote 5@M. Yeadon, M. T. Marshall, F. Hamdani, S. Pekin, H. Morkoc, and J. M. Gibson, Journal of Applied Physics 83, 2847 (1998).

4:20pm **TF-TuA8 Direct Three-Dimensional Characterization of Buried Interface Morphology with Quantized Electron Waves**, *I.B. Altfeder,* ***D.M. Chen***, The Rowland Institute for Science

We present a novel in situ and nondestructive technique for characterizing buried interfaces in metal/semiconductor heteroepitaxy. The principle of the technique is based on the quantum confinement of the internal electron source in the metal film. Due to the high sensitivity of the quantized electron waves to the boundary conditions, discrete interference fringes are formed spontaneously on the surface of the film and can be directly imaged by a scanning tunneling microscope.@footnote 1@ These fringes coincide precisely with the substrate atomic terraces so that the metal appears to be "transparent". The absolute depth of the film, on the other hand, can be accurately determined form the tunnel I-V measurements of the quantized energy spectra near the Fermi level, hence making it possible to characterize the buried interface morphology in all three dimensions nondestructively. @FootnoteText@ @footnote 1@I. B. Altfeder, K. A. Matveev, and D. M. Chen, Phys. Rev. Lett. 78, 2815 (1997).

4:40pm **TF-TuA9 A Novel Design of a Reflecton Analyzer for Elemental and Isotopic Analysis by MSRI (Mass Spectroscopy of Recoil Ions)@footnote 1@**, ***K.L. Waters***, *K. Baudin, J.A. Schultz,* Ionwerks

Mass Spectroscopy of Recoil Ions (MSRI) is a recently developed technique which has been reviewed@footnote 2@ and has been used in real time monitoring of nitride@footnote 3@ and oxides growths. The technique is similar to Time of Flight SIMS (TOF/SIMS) in that a pulsed keV primary ion beam is impinging at grazing incidence onto the analyzed surface. Sputtered ions are collected and analyzed by TOF. By careful design of the extraction optics using differential pumping and by placement of the analyzer in the forward scattering angle at around 60-70 degrees, it is possible to achieve mass spectra of directly recoiled binary ions which are devoid of molecular interference at pressures in excess of 1mTorr. For the optic positioned at 1inch from the focal point of the primary ion beam onto the sample, Mo isotopes have been resolved with a resolution at half maximum of 450. Operation at 2 inches from the focal point reduces the resolution to 250 and reduces the collection and transmission of recoiled ions by a factor of two. Examples will be given of the use of the MSRI technique for both exsitu and real time control of deposition process. @FootnoteText@ @footnote 1@Financial support of this work by US Air Force SBIR Contract F33615-970C-1035 @footnote 2@M. S Hammond, J.A Schultz, A. R. Krauss J.Vac.Sci. Technol. A 13(3) 1995 @footnote 3@E. Kim, I. Berishev, A. Bensaoula, S. Lee, S.S. Perry, K. Waters, J.A. Schultz Appl. Phys. Lett. 71 (21) 1997

5:00pm **TF-TuA10 The Interaction of Al Atoms with Surface-Bound Organic Functional Groups Studied by In-situ XPS, Infrared Spectroscopy and ToF-SIMS**, ***A.E. Hooper***, *G.L. Fisher,* Pennsylvania State University*; R.L. Opila,* Bell Laboratories, Lucent Technologies*; N. Winograd, D.L. Allara,* Pennsylvania State University

The interaction of vapor-deposited Al atoms with self-assembled monolayers of structure HS(CH2)15CH3 and HS(CH2)15COOCH3 was studied with multiple in-situ techniques over a range of Al coverages from submonolayer to multilayers. XPS and IR show the Al to be unreactive with the CH2 units but highly reactive with the oxygen atoms in the ester group to form a 1:1 Al:ester bridged type of complex with an intact CH3 group and a loss of double bond character for the C=O group. In the case of the CH3-termimnated surface, ToF-SIMS shows the Al atoms penetrate into the organic monolayer and diffuse to the Au-S interface. Both XPS and ToF-SIMS indicates that subsequent reaction with the S atoms appears to occur,

but in such a way as not to disturb the packing of the monolayer hydrocarbon chains. A dynamic fluctuation mechanism is proposed to explain the Al atom penetration in the CH3 case. In the ester-terminated film, penetration appears to be thwarted by the rapid reaction of the Al atoms with the ester terminal groups to form a dense monolayer of organometallic species.

## Thin Films Division
## Room 310 - Session TF-WeM

### ULSI Metalization and Interconnects

**Moderator:** J. Hopwood, Northeastern University

8:20am **TF-WeM1 ULSI Metallization and Interconnects**, ***C.B. Whitman,*** CVC, Inc. **INVITED**

The performance of advanced IC chips is limited by the interconnect propagation delays, dispersion, and cross-talk within the Al/SiO@sub 2@ system. The combination of Cu and low-k dielectrics enables a reduction of the interconnect levels and improved reliability. Various deposition methods (MOCVD, PVD, and plating) have been evaluated for Cu. Vacuum-integrated clustering of single-wafer process modules offers advantages for advanced metallization applications. The capability to integrate the pre-clean process with the copper (seed and/or fill) and barrier layers in a vacuum cluster tool prevents atmospheric exposure of the sensitive metallization interfaces, resulting in improved process repeatability and reduced via resistance. A vacuum-integrated MOCVD metallization technology has been developed and demonstrated for formation of high-performance Cu plugs/lines for 0.18 - 0.13 µm IC manufacturing. MOCVD copper seed and gap-fill processes have been integrated with soft plasma clean and barrier deposition processes in a vacuum cluster tool. The MOCVD-Cu process is capable of void-free filling of high-aspect-ratio (up to 8:1) features with low-resistivity (@<=@2 micro-ohm.cm) Cu layers. Excellent copper layer adhesion and copper/barrier microstructure properties have been achieved. The films have shown negligible impurities and large (0.5 - 0.8 µm) grains with texturing. Deposition rates in the range of 400 to 3000 Å/min have been achieved. Inlaid MOCVD copper lines and vias have been fabricated with TiN and TaN barrier layers using CMP damascene processing. MOCVD-Cu offers performance advantages for fabrication of void-free inlaid lines/plugs for all the interconnect levels, including the lower levels with narrower and larger aspect-ratio lines and the upper interconnect levels with wider/thicker and smaller aspect-ratio lines. We have also demonstrated successful process integration of copper electroplating via/trench filling with MOCVD cluster tool barrier and seed formation.

9:00am **TF-WeM3 Pinhole Formation in Solid Phase Epitaxial Film of CoSi@sub 2@ on Si(111)**, ***L. Ruan,*** *D.M. Chen,* The Rowland Institute for Science

We have revisited the long-standing pinhole problem in solid phase epitaxial growth of a CoSi@sub 2@ film on Si(111) with in situ scanning tunneling microscopy.@footnote 1@ While the as-deposited film with 5 Å of Co at room temperature shows a smooth granular texture with original substrate terraces remaining intact, annealing at 580ºC produces an epitaxial CoSi@sub 2@ film with large pinholes enclosed by a thin ring CoSi@sub 2@, exhibiting a volcano feature. Quantitative analysis shows that the formation of pinholes is a result of rapid Si outward diffusion from bulk to surface, and of the subsequent Si reaction with Co on the outer surface. Evidence suggests that inhibiting the Si diffusion channels during the thermal annealing process is the key to solving the pinhole problem. @FootnoteText@ @footnote 1@Like Ruan and D. M.Chen, Apply. Phys. Lett. in press.

9:20am **TF-WeM4 Investigation of the Structural and Chemical Stability of Advanced Metal Gate and Ultra-Thin Gate Dielectric Interfaces**, ***B. Claflin,*** *G. Lucovsky,* North Carolina State University

Aggressive scaling of MOS device features to 0.1 µm and below will require the introduction of novel materials and new, low-thermal-budget processes. For example, in front end-of-the-line processing, alternative gate dielectrics such as ultra-thin SiO@sub 2@/Si@sub 3@N@sub 4@ stacks and/or high dielectric constant (K) materials such as Ta@sub 2@O@sub 5@ will be required to scale down the oxide equivalent thickness, t@sub ox@, and limit leakage current to acceptable levels. Likewise, replacements for heavily doped polycrystalline Si (poly-Si) gate electrodes such as simple or compound metals will be needed to prevent i) poly-depletion and ii) boron out-diffusion and dielectric penetration effects. However, the compatibility of these new materials under device processing conditions must be demonstrated. For example, many transition metals chemically react@footnote 1,2@ with SiO@sub 2@ at temperatures above 650@degree@C, and as such can not be used in gate stacks. In addition, the effects of thin film microstructure or phase transitions can dramatically alter the electronic and mechanical properties of these materials, and their interfaces, degrading both device performance and reliability. Recent studies@footnote 3,4@ indicate that TiN@sub x@ and WN@sub x@ composite metal gates are compatible with ultra-thin remote plasma-enhanced chemical-vapor deposited (RPECVD) SiO@sub 2@ and stacked SiO@sub 2@/Si@sub 3@N@sub 4@ gate dielectrics that are subjected to rapid thermal annealing (RTA); i.e., they perform well as metal gate electrode in MOS device structures. In this work, the structural integrity of these metal/dielectric interfaces subjected to RTA is investigated by SEM, TEM, and X-ray diffraction. These structural characterizations are correlated with the chemical stability of these interfaces determined by Auger electron spectroscopy (AES). Supported by NSF, SRC, and ONR. @FootnoteText@ @footnote 1@ S. Q. Wang and J. W. Mayer, J. Appl. Phys. 64, 4711 (1988). @footnote 2@ R. Pretorius, J. M. Harris, M-A. Nicolet, Solid State Electron. 21, 667 (1978). @footnote 3@ B. Claflin, M. Binger, and G. Lucovsky, J. Vac. Sci. Technol. A 16 (1998), in press. @footnote 4@ B. Claflin, M. Binger, and G. Lucovsky, Mat. Res. Soc. Symp. Proc. (1998), in press.

9:40am **TF-WeM5 Low Temperature Deposition of Zirconium Diboride, A Candidate Diffusion Barrier, Using Remote Plasma CVD**, ***J.H. Sung,*** *D.M. Goedde, G.S. Girolami, J.R. Abelson,* University of Illinois, Urbana-Champaign

Zirconium diboride is potentially suitable as an advanced diffusion barrier in ULSI circuits because of its low electrical resistivity, 10 micro-ohm-cm in bulk form, very high melting temperature, ~ 3000 C, and resistance to air oxidation and reaction with metals. However, the CVD of ZrB2 from conventional halide/hydrogen source gases requires a relatively high temperature of ~ 900 °C. We showed that zirconium tetrahydroborate, Zr(BH@sub 4@)@sub 4@, is an attractive precursor which has high vapor pressure and can be thermolyzed at < 300 °C to produce ZrB2 films with resistivity ~ 120 µm@OMEGA@-cm. In this work, we report the use of remote plasma CVD to further reduce the ZrB2 deposition temperature and improve the film properties. Atomic hydrogen is generated by a microwave plasma source and mixed downstream with Zr(BH@sub 4@)@sub 4@ to produce high quality films at only 150 °C. The films have resistivity ~ 60 µm@OMEGA@-cm, and low carbon and oxygen contamination. XPS data indicate that the plasma-deposited films are single phase ZrB2. Based on in-situ mass spectroscopy data, we will present a preliminary analysis of the chemical reaction pathways associated with the remote plasma growth process.

10:00am **TF-WeM6 A Parameter Free Model for the Simulation of Trench Filling Profiles under Al PVD and Al IPVD Conditions**, ***A. Kersch,*** Siemens Ag, Germany*; U.P. Hansen,* Technical University Munich, Germany

Simulation results are presented for microscopic profile evolution of deposited Al metal films in trench structures. The model for the simulation is derived from atomistic, molecular dynamics calculations using a previously developed Al-Al interaction model.@footnote 1@ The essential elements are: (1) an angular and energy dependent non unity sticking coefficient resulting in specular reflection of the impinging Al atoms, (2) an energy and angular dependent sputter yield. The parameters of the model are obtained from the molecular dynamics results, surface diffusion is so far neglected. We study the surface evolution and sidewall coverage for different PVD and IPVD conditions for trench structures of different aspect ratios and clarify the influence of the model elements on the deposition process. The predictions of the model agree with the results of a published model@footnote 2@ in some range of process conditions. Finally results are compared to experimental data. @FootnoteText@ @footnote 1@U. Hansen, Molecular Dynamics Study of Al PVD Processes @footnote 2@S. Hamaguchi and S.M. Rossnagel, J. Vac. Sci. Technol. B13 (1995) 183

10:20am **TF-WeM7 Energy Dependent Atomistic Simulations of Trench-filling**, ***Y.G. Yang,*** *X.W. Zhou, H.N.G. Wadley,* University of Virginia

A comprehensive atomistic analysis of incident kinetic energy effects during vapor deposition has been conducted and the results integrated into a kinetic Monte Carlo simulation of physical vapor deposition. Interactions of hyperthermal atoms with substrate that resulted in biased diffusion, atomic reflection, resputtering and local thermal spikes were incorporated in a simulation model that also included normal, thermally driven multipath diffusional processes. Results are presented for the vapor phase deposition of metal interconnects by the dual damascene process. This involves the filling of increasingly narrow trenches with copper and other metals. Filling of these trenches has been studied as a function of the substrate temperature, the deposition rate, trench geometric parameters, incident atom flux energy and angular distributions.

10:40am **TF-WeM8 Sputtered Copper Seedlayer Processing Issues**, ***E.C. Cooney III,*** *D.C. Strippe, J.W. Korejwa, A.H. Simon, C. Uzoh,* IBM Microelectronics

One method to produce reliable Copper interconnects for advanced logic chip technology utilizes sputtered Copper seedlayers which are subsequently electroplated. However, experimental results indicate that good film step coverage as well as conformality are necessary to promote complete filling of the feature. Current trench dimensions and dual damascene aspect ratios are such that traditional sputter processes cannot adequately cover these structures. In addition, Copper tends to dewet from the substrate when thermal aspects of the process, such as sputter energy and deposition temperature, become too great. This can lead to a discontinuous film causing void formation during electrodeposition. We have investigated collimation of Copper films to improve the conformality in aggressive single and dual damascene structures. Collimation filters of various aspect ratio were used to deposit Copper seedlayers which were then filled using electrodeposition. In addition, thermal effects were examined through experimentation with lower DC magnetron powers coupled with special platen cooling considerations to reduce the heat load within the film. Electrical opens-yield and resistance data was then measured. Failure analysis was also performed to observe the plating fill.

11:00am **TF-WeM9 The Effect of Sputter Process and Target Pass-Through Flux on Sputter Deposition of Co Thin Film for Cobalt Silicide Metallization**, ***H. Zhang***, *J. Poole, R. Eller, M. Keefe,* Tosoh SMD, Inc.

CoSi@sub 2@ is considered an alternative to TiSi@sub 2@ for use as a contact in ULSI due to its low resistivity, excellent chemical stability and lower formation temperature. Sputter deposition of Co thin film is one of the crucial steps in salicide (self-aligned silicide) process. One major problem with sputter deposition of Co thin film, however, is that Co is a ferromagnetic material, which can be difficult to sputter. Magnetic field strength has been found to be the key parameter in sputtering magnetic films. In magnetron sputtering of Co, high magnetic field strength can be obtained by using a high pass-through flux (PTF) target that allows maximum magnetic flux from the magnets to permeate through the magnets. A high PTF target also allows efficient sputtering and uniform target erosion. The effects of target PTF and sputtering process parameters such as Ar pressure, sputtering power, target to substrate spacing and substrate temperature on the sputter process and film properties were studied. Co targets with PTF of 65% (high PTF), 55% (medium PFT) and 35% (low PTF) were sputtered. Co thin films ranging from 16 nm to 500 nm in thickness were deposited on 200 mm (100) Si wafers. Sputter deposition rate, I-V characteristics, film sheet resistance and film uniformity was measured under various sputter conditions. It is indicated that the I-V characteristics of different PFT targets followed the normal I=KV* relationship of DC planar magnetron sputtering. The exponents in the equation, n, increased with increasing target PTF, indicating the higher the PTF, the lower the impedance. The target having the highest PFT demonstrated the best film uniformity. Rapid thermal processing was carried out to form cobalt silicides at temperatures between 300°C to 850°C in Ar ambient for various times. The sheet resistance of the Co and cobalt silicide films was monitored by four-point probe before and after the RTP. Phases and microstructure of the films were characterized using XRD, SEM and SIMS. The sheet resistance decreased significantly after annealing at 600°C and 700°C due to formation of CoSi@sub 2@, which has lower resistivity. The significant increase in sheet resistance after annealing at 400°C and 500°C was attributed to formation of CoSi phase. For the 16 nm thick Co film, sheet resistance of 3.2 @OMEGA@/sq was obtained after RTP at 600°C and 700°C.

11:20am **TF-WeM10 Improvement of Morphological Stability of Ag Thin on TiN Layer**, ***C.-Y. Hong***, Massachusetts Institute of Technology*; Y.-C. Peng, L.-J. Chen,* National Tsing-Hua University, Republic of China*; W.-Y. Hsieh,* United Microelectronic Corporation, Republic of China

Owing to the need to increase the packing density in ULSI, thermally stable low-resistive contact and metallization technologies are important. Numerous studies of noble-metal-semiconductor processes have been conducted to obtain a better understanding of the interface structure and other properties. Ag is the most conductive materials and has been considered to be a candidate materials for interconnection in ULCI fabrication. Since fast interdiffusion occursbetween Ag and Si substrate, advanced metallization technologies of Ag for ULSI require a highly comformal barrier layer to prevent the interdiffusion between Ag and Si substrate. Among various kinds of diffusion barriers, titanium nitride (TiN) thin films have been widely used in ULSI fabrication due to its relatively low electrical resistivity and high thermal stability. In a previous study, Ag islands were found to form on TiN layers after annealing at 100 °C for 30 min. Owing to the poor morphological stability of the Ag/TiN interface, the utilization of thin Au and Ti layers between Ag and TiN layers has been explored to overcome the island formation problem in this study. The presence of interposing Au and Ti layers was found to increase the morphological stability temperature of Ag thin films on TiN layer from 100 °C to 450 °C and 350 °C, respectively.

## Thin Films Division
## Room 310 - Session TF-WeA

### Advances in Sputtering

**Moderator:** S. Zarrabian, Optical Coatings Laboratories, Inc.

2:00pm **TF-WeA1 Preferential Sputtering Effects in Thin Film Processing**, ***S. Berg***, *I.V. Katardjiev,* Uppsala University, Sweden **INVITED**

Predicting the partial sputtering yield (number of sputtered atoms of one element per one incident ion) for the different constituents during sputtering from a multielemental surface is a rather difficult task. For an alloy bulk target where no diffusion takes place, however, it can at least be assumed that the ratio of the outsputtered elemental fluxes is exactly equal to the corresponding composition ratio of the target bulk. During bias sputter deposition from an alloy target, however, the composition of the deposited film may deviate strongly from the target composition due to preferential re-sputtering of one (or more) elements from the growing compound film. We will present a systematic study that serves to clearify how and why some atomic elements are preferentially sputtered from a multielement matrix. By using a Monte Carlo based computer simulation program (T-DYN) it is possible to simulate the evolving collision cascades in the bombarded material and thus study the sputtering process in its dynamics. The results from such computer simulations indicate that the partial sputtering yield of one element in a multielement matrix depends in a systematic way on the atomic density of the material, the atomic number of the atomic elements and the projected range of the incoming energetic ion. Furthermore, from this study we have found out that it is possible to "tune" the partial sputtering yield of one element in a multielement matrix. The "tuning" effect can be quite dramatic. Adding a few percent of e.g. W to a pure Al target may increase the Al sputtering yield by as much as 100% as compared to the sputtering yield of pure Al. Other interesting effects caused by "controlled preferential sputtering" will also be demonstrated.

2:40pm **TF-WeA3 Measurements and Modeling of Ti and Ta Sputtering as a Function of Target Microstructure and Temperature**, ***J.P. Allain***, *D.A. Alman, D.N. Ruzic,* University of Illinois, Urbana-Champaign

The angular distribution of sputtered material and the absolute sputtering yield of metal targets by argon ions at energies less than 1000 eV has been measured in previous work for a number of materials.@footnote 1@ The use of titanium and tantalum films are continually applied as effective underlayers for both barrier and enhanced metallization properties. This paper focuses on the influence of target microstructure and temperature on the sputtering and angular distribution yields. A Colutron ion gun is used to produce an ion beam which is decelerated near the target. The beam diameter near the target is modified so as to focus on a single grain. The beam diameter can also cover several grains including grain boundaries. Grain boundary density and orientation is studied on its effect on the distribution and yield. The diagnostics near the target can be rotated to intercept the sputtered flux. The diagnostics consist of a quartz crystal oscillator to measure total yield and a cylindrical pyrolytic graphite collector plate. The graphite plate is analyzed by an PHI Auger spectrometer to obtain areal densitites and thus the angular distributions. The target assembly is fixed and monitored by a thermocouple. A "cold finger" which can deliver liquid N2 is attached to the target. Modeling of the system is used by an enhanced version of VFTRIM3D, a code which includes fractal geometry and a non-binary collision model.@footnote 2@ @FootnoteText@ @footnote 1@W. Eckstein, C. Garcia-Rosales, J. Roth, W. Ottenberger, "Sputtering Data", pub. Max-Planck-Institut Fur Plasmaphysik, February 1993. @footnote 2@D.N. Ruzic, Nucl. Instrum. Methods B 47 (1990) 118.

3:00pm **TF-WeA4 Reactor-Scale Models for Rf-Diode Sputtering for GMR Thin-Film Growth**, ***S. Desa***, *S. Ghosal, R.L. Kosut, J.L. Ebert, A. Kozak, T.E. Abrahamson,* SC Solutions*; J.F. Groves, H.N.G. Wadley, D.W. Zou,* University of Virginia

This paper describes the development of a physical model for the Rf-diode sputtering of GMR thin-films. The model consists of: (1) a CFD finite element model for the velocity and pressure distribution of the Argon (Ar) gas flow in the chamber, (2) a steady-state plasma model for the flux and energy of Ar ions striking the target and the substrate, (3) a molecular dynamics (MD) sputtering model for the energy distribution, angle distribution, and yield of the Copper (Cu) atoms sputtered from the target by the Ar ions, and (4) a Direct Simulation Monte Carlo (DSMC) model for the transport of Cu atoms through the low-pressure argon gas to the deposition substrate. The individual models for gas flow, plasma discharge, Cu sputtering, and DSMC-based Cu atom transport are then integrated to create a detailed, steady-state, input-output model capable of predicting thin-film deposition-rate and uniformity as a function of the process input variables: power, pressure, gas temperature and electrode spacing. (Deposition and uniformity in turn define well-known device characteristics such as H@sub sat@ and GMR ratio.) The paper also describes the development of an approximate input-output model whose CPU time is several orders-of-magnitude faster than that of the detailed model. Both models were refined and validated against experimental data obtained from an actual GMR chamber.

3:20pm **TF-WeA5 Using Pulsed DC Power for Reactive Sputtering of Al@sub 2@O@sub 3@**, ***A. Belkind***, *A. Freilich,* Stevens Institute of Technology*; R. Scholl,* Advanced Energy Industries

Implementation of reactive sputtering of dielectrics such as Al@sub 2@O@sub 3@ by the use of steady DC power is obstructed by arcing. The arcing appears to be due to breakdown of the dielectric (oxide) films that grow on the metal target surface and which accumulate positive charges on their surfaces due to ion bombardment. The arcing can be greatly alleviated when pulsed DC power is applied. By pulsed DC power we mean that the power is applied for a short "on" period, and then removed for a short "off" period. During the "off" period the plasma can discharge the surfaces, provided certain conditions are met. The dependence of adequate discharging, and thus arc prevention, on the duration of the "on" and "off" periods is examined. In addition, the dynamics of plasma density loss in the "off" period and its re-establishment in the initial part of the "on" period are discussed. Reactive sputtering takes place only during the "on" period, and part of this period is lost for effective sputtering due to the necessity for full plasma re-establishment. This produces a dependence of the deposition rate on both the duty cycle and the frequency of pulsing, but not on the power. This dependence is examined and the power efficiencies of AC and pulsed power DC reactive sputtering are compared and speculation made as to the differences in results published between single and dual cathode systems.

3:40pm **TF-WeA6 Suppression of Hillocks and Whiskers on Al Films Deposited onto a Glass**, ***H. Saka***, *Y. Suzuki,* Nagoya University, Japan*; H. Takatsuji, K Tsujimoto,* IBM, Japan*; K. Kuroda,* Nagoya University, Japan*; S. Tsuji,* IBM, Japan

A new technique to suppress formation of hillocks and whiskers on an Al film deposited onto a glass substrate has been developed. First, an Al film was deposited on a LCD-grade glass substrate by industrially conventional dc magnetron sputtering. Heating this films above 573K resulted in formation of a number of hillocks and whiskers. Onto this Al film another layer of Al film was deposited by rf magnetron sputtering. When the thickness of the second Al layer is very thin, many hillocks and whiskers are formed on heating. However, when the thickness of the second Al layer is around 40nm, the formation of both hillocks and whiskers are completely suppressed. Cross-sectional TEM observation revealed that the second layer of Al deposited by rf sputtering is amorphous. The sheet resistance of the second layer was measured by van der Pauw method to be 3.5 x 10@super-6@ ohm cm.

4:00pm **TF-WeA7 Substrate Bombardment and Heating in Dual Magnetron Sputtering Using Mid-Frequency AC**, ***J. Plaisted***, Kinneo*; G.W. McDonough, G.A. Roche,* Advanced Energy

Dual magnetron sputtering (DMS) using mid-frequency AC has become a popular method for the reactive deposition of dielectrics. However, several studies have reported higher levels of substrate bombardment and heating with the use of this technique. In an effort to determine the cause, we separated the effects of plasma ignition from those of the anode arrangement. The anode arrangement employed in mid-frequency DMS was duplicated in DC operation using a pair of opposed magnetrons powered by a floating output generator. Values of the substrate self-bias, ion current, and temperature were compared to those obtained from running the sources with 40 kHz AC and standard DC techniques. Results indicate that a large fraction of the energetic species found in mid-frequency DMS can be explained by a restriction of the anode surfaces to the paired magnetrons.

4:20pm **TF-WeA8 A Novel Approach to Collimated Physical Vapor Deposition**, *A.P. Paranjpe,* ***D. Heimanson***, *J.C.S. Kools, P.V. Schwartz, K. Song, B. Bergner, S. McAllister,* CVC

In some applications of Physical Vapor Deposition (PVD), it is desirable to have the atoms arriving at the substrate at angles close to the normal

(collimation). Applications of collimated PVD include filling of highaspect ratio vias, as used in multilevel IC metallization, or high resolution shadow masking as used in lift-off technology. Several approaches to collimation have been proposed in recent years: natural (or long throw) collimation, physical collimation, ion beam deposition ionized PVD and hollow cathode collimation. In this contribution, we introduce a novel approach to collimation. It is found experimentally that this approach leads to a strongly improved degree of collimation when compared to the conventional collimation methods. Contrary to ion beam deposition and ionized PVD, our approach allows to vary the kinetic energy of the sputtered atoms arriving at the substrate over a wide range. Simulations of the target-to-substrate atom transport using the Simbad package show that the improved collimation method leads to angular distributions with Full Widths at Half Maximum (FWHM) below 20 degrees.

4:40pm **TF-WeA9 Microcrystalline Silicon Thin Films Deposited By Low Temperature Reactive Magnetron Sputtering: The Effect Of Using Deuterium vs. Hydrogen**, ***J.E. Gerbi,*** University of Illinois, Urbana-Champaign*; D.S. Kim,* SAIT, Korea*; G. Ben Amor,* Ecole Polytechnique, France*; J.R. Abelson,* University of Illinois, Urbana-Champaign

Microcrystalline silicon (uc-Si:H) thin films are of interest for macro-electronic technologies: they can serve as optical absorber or doped contact layers in solar cells, or as the nucleation layer in the direct deposition of polycrystalline silicon on glass for thin film transistors.@footnote 1@ The grain size, shape, and defect density significantly modify the electronic properties of uc-Si:H; therefore, it is highly desirable to control the film microstructure through the growth process. We previously showed that DC reactive magnetron sputtering (RMS) of a Si target produces uc-Si:H films when sufficient H@sub 2@ is added to the Ar working gas. In the sputtering plasma, H@sub 2+@ ions are accelerated towards the Si target and reflect as fast neutral H atoms, which impinge on the growing film and implant to a depth of ~ 50Å. This large flux of fast H atoms provides unique control over the nucleation and growth of the uc-Si:H phase.@footnote 2@ In this work, we explore the effects of using D@sub 2@ instead of H@sub 2@ to grow uc-Si:H films on glass at a substrate temperature of 230°C. The substitution of D@sub 2@ for H@sub 2@ lowers the partial pressure at which the microcrystalline regime is entered, and produces films with a higher degree of crystallinity throughout the entire pressure range investigated. Crystalline nucleation and the grain-size dependent electronic structure are observed in real time using spectroscopic ellipsometry. We report post-deposition TEM, Raman spectroscopy, and electrical characterizations. To explain the implantation-related isotope effect, we present binary collision (TRIM) simulations of the energy distribution, range, and recoil behaviors of the H vs. D neutral fluxes, and their resultant dynamic concentrations in the film. @FootnoteText@ @Footnote 1@Y. H. Yang and J. R. Abelson, Appl. Phys. Lett. 67, 3623 (1995). @Footnote 2@Y. H. Yang, M. Katiyar, N. Maley, and J. R. Abelson, Appl. Phys. Lett. 65(14), 1769 (1994).

5:00pm **TF-WeA10 Atomistic Simulations of the Sputter Deposition of Copper**, ***W. Zou,*** *J.F. Groves, X.W. Zhou, H.N.G. Wadley,* University of Virginia

RF Diode sputter deposition is being explored for synthesizing metal and magnetic multilayer films. A discrete simulation Monte Carlo binary collision model has been developed to analyze the spatial uniformity and impact velocity of individual vapor atom with a substrate. The analysis began with the calculation of ion-impact with a metal target using Molecular Dynamics simulations. This provided both the initial energy distribution of vapor atom and its angular distribution immediately upon their sputter emission from the target. The model then used input conditions such as background pressure, temperature, gas type, and reactor geometry in combination with a discrete simulation Monte Carlo method to analyze vapor atom transport to the substrate. Results are shown of vapor atom deposition efficiency, the spatial distribution of the film thickness, the impacting atom energy and the impact angle distribution of the vapor atoms. These vapor transport model results provide a link between the microstructure of thin films and the deposition process conditions, and can be used for reactor design and control.

## Thin Films Division
## Room 310 - Session TF-ThM

### Thin Films for Flat Panel Applications

**Moderator:** G.N. Parsons, North Carolina State University

8:20am **TF-ThM1 New Dry Etch Applications for Amorphous TFTs in Flat Panel Displays(FPD)**, ***W.W. Yao,*** dpiX; A Xerox New Enterprise Company **INVITED**

Key goals driving FPD process development are: 1. large panel size; 2. Low cost; 3. Low power consumption. Recent introduction of new process technologies especially in the dry etch area are key enablers for new display architecture. Increase in display size with longer gate and data lines is driving the switch to aluminum metal with its lower resistivity to reduce the RC time delay. Hillock free Aluminum gate metal is enabled by tapered Al dry etching and high rate PECVD process with short time-temeprature cycle. Cost reduction has focused on productivity improvement but new 3rd generataion tools has renewed interest recently in reduced mask count display architecture. ITO pixel is etched using very strong acids and dictates its placement in the process architecture. New ITO dry etch removes process constraints and allows placement of the ITO on top of the passivation dielectric. Power consumption is dominated by backlight intensity and can be reduced by higher aperture ratio pixel design. The ITO pixel dimension is increased if the TFT size and its assoicated coupling capacitance is shrinked through the use of selective n+ etch in intrinsic silicon.

9:00am **TF-ThM3 Field Emission and Photo Emission from Si Micro Tip Arrays Coated with Bias-Grown Diamond Films**, ***M.Q. Ding,*** *A.R. Krauss,* Argonne National Laboratory*; O. Auciello,* Argonne National Laboratory, U. S. A.*; D.M. Gruen, T.D. Corrigan,* Argonne National Laboratory*; M.E. Kordesch,* Ohio University*; D. Temple, D. Palmer, G.E. McGuire,* MCNC

A considerable improvement in the properties of field and photoelectron emission from ungated Si micro tip arrays coated with bias-grown diamond thin films is reported. Prior to loading into a MPCVD reactor (ASTeX PDS-17), the microtip arrays were ultrasonically treated in a 0.1 µmm diamond suspension. Diamond films were grown in a CH@sub 4@-H@sub 2@-N@sub 2@ plasma at a substrate temperature of 800 °C and a negative bias of -150 V. The film had a complete coverage over the tip arrays as observed from secondary electron microscopy (SEM). Electron emission characteristics were measured in two different systems: field emission current-field (IF) measuring apparatus and photoelectron emission microscopy (PEEM). IF measurements showed a very low turn-on electric field with a threshold fields of 1.5 V/µmm (vs 40 V/µmm for uncoated tip arrays), and a current density of 1 mA/cm2 at around 4 V/µmm. In the PEEM studies, UV light from a mercury arc lamp was used to excite photoelectrons. While the lamp was on, the PEEM revealed a uniform and bright photoelectron emission image of the coated tip arrays in contrast to that of the uncoated arrays. When the lamp was off, stable field electron emission images of the coated tip arrays were also seen with a reasonable brightness at an electric field 5.6 V/µmm, whereas images of uncoated arrays could hardly be discerned and emission was unstable. Such a remarkable improvement in both field emission and photoemission properties, as compared to those of the uncoated arrays, indicates that the microtip arrays coated with bias-grown diamond films are promising for applications such as flat panel displays. A possible mechanism for the improvement will be discussed. This work is supported by the U.S. Department of Energy, BES-Materials Sciences, under Contract W-31-109-ENG-39.

9:20am **TF-ThM4 Hydrogen Concentration Distribution in Plasma Deposited Hydrogenated Amorphous Silicon and Silicon Nitride Films**, ***B.F. Hanyaloglu,*** *D.C. Marra, E.S. Aydil,* University of California, Santa Barbara

Understanding H distribution in plasma deposited hydrogenated amorphous silicon and silicon nitride (a-SiN:H) films as well as at the interfaces of these films is important for manufacturing of thin film transistors (TFTs) for flat panel display applications. Hydrogen concentration distribution and bonding in plasma deposited a-Si:H and a-SiN:H thin films were studied using in situ multiple internal reflection Fourier transform infrared spectroscopy in conjunction with in situ spectroscopic ellipsometry. The infrared spectra as a function of time were recorded both during deposition of the film and during etching with CF@sub 4@ plasma. The hydrogen concentrations as a function of depth below the film surface were obtained from the spectra recorded during the deposition and the etching experiments. Analysis of the spectra shows that the H distribution in a-Si:H and a-SiN:H films is surprisingly complex and far from uniform. The a-Si:H film consists of a very thin H rich layer at the surface that is primarily composed of SiH@sub 2@ and SiH@sub 3@. This H-rich surface layer is followed by a few 100 Angstrom thick subsurface region that is depleted in H compared to the bulk film. The bulk a-Si:H film grows beneath these two layers, which move up and stay at the surface during deposition. In a-Si:H deposition, there is evidence that H penetrates into the film through a process other than simple diffusion. We have also investigated H distribution and bonding in a-SiN:H films deposited on a-Si:H and a-Si:H films on a-SiN:H in order to study how the deposition sequence alters the chemical composition and structure of the a-Si:H/a-SiN:H interfaces. Implications of these experimental results on the differences in the peformance of top-gate and bottom-gate configuration TFTs will be discussed.

9:40am **TF-ThM5 Poly-Si Thin Film Transistors Fabricated on Low Temperature Plastic Substrates**, ***P.G. Carey,*** *P.M. Smith, P. Wickbolt, S.D. Theiss,* Lawrence Livermore National Laboratory **INVITED**

Flat panel displays made on plastic substrates are envisioned for use in certain commercial and military systems because they are more rugged and lightweight than displays made on glass substrates. High information content can be attained for such displays using active matrix arrays of thin film transistors (TFTs). In this talk the fabrication of poly-Si TFTs on flexible plastic substrates will by discussed. Plastic substrates pose severe temperature constraints on the fabrication process. To overcome these constraints, our group at LLNL has used low temperature (<150C) silicon, oxide, and aluminum thin film deposition steps and pulsed excimer laser processing to perform the TFT channel crystallization and the source/drain doping.

10:20am **TF-ThM7 Stability of Very Low Temperature Amorphous Silicon Thin Film Transistors on Flexible Plastic Substrates**, *C.S. Yang,* ***L.L. Smith,*** *C.B. Arthur, G.N. Parsons,* North Carolina State University

Active matrix transistor arrays on transparent plastic substrates will enable new high resolution flexible and rugged large area electronic display systems, including liquid crystal displays (LCDs), and organic light emitting displays (OLEDs). Hydrogenated amorphous silicon thin film transistors (TFTs) for active matrix LCDs are currently formed on glass substrates using temperatures in excess of 250°C. Lower temperature processes are of interest for TFTs on plastics, but stability of low temperature TFTs has not been reported. In this presentation, we will describe low temperature (0.3 cm@super 2@/V-s and off currents

10:40am **TF-ThM8 Advanced Deposition Technique for Producing Thin Films of Polycrystalline Silicon**, ***J.B.O. Caughman,*** *D.B. Beach, G.L. Bell,* Oak Ridge National Laboratory

An improved plasma enhanced chemical vapor deposition technique has been demonstrated for depositing poly-crystalline silicon thin films for flat panel display applications. The technique combines a high density radio frequency (rf) inductively coupled plasma source with downstream gas injection that has resulted in device quality films deposited at high rates. Unlike conventional rf reactors, inductively coupled sources have high plasma and atomic species density along with low ion energies hitting the film surface during growth. Our system uses a planar induction coil for the plasma coupling that creates a dense hydrogen plasma over a large area (30 cm diameter). The hydrogen plasma serves as a source of large quantities of atomic hydrogen that aid in the deposition process. For better control of the plasma chemistry, silane (100%) is injected downstream, where the precursors needed for film growth are separated from the ionization region. The films are deposited on quartz samples on a heated substrate (< 400 degrees C). The power coupling mechanism (inductive vs. capacitive coupling) has been analyzed by using an rf sensor (located after the matching network) to determine processing conditions favorable for polysilicon growth. Deposition rates increase substantially with the amount of inductive power coupling and reach values of 60-80 nm/min. Increasing the coupled power beyond 1 kW eventually leads to a decrease in the net deposition rate, possibly due to increased etching of the deposited film by the hydrogen. Deposition results show that the conductivity (10@super -7@ S/cm) and the crystallinity (>80% based on the Raman spectrum) of the films are good. Analysis of the X-ray diffraction spectrum shows a highly preferred grain orientation in the  plane. Details of the deposition conditions and the power coupling mechanism will be discussed. @FootnoteText@ Research sponsored by the Laboratory Directed Research and Development Program of ORNL, managed by Lockheed

Martin Energy Research Corp. for the U.S. Department of Energy under contract no. DE-AC05-96OR22464.

11:00am **TF-ThM9 Polycrystalline Silicon Films Deposited Directly on Glass by Reactive Magnetron Sputtering Using a Microcrystalline Silicon Nucleation Layer**, ***D.S. Kim,*** Samsung Advanced Institute of Technology, Korea*; J.E. Gerbi, J.R. Abelson,* University of Illinois, Urbana-Champaign

We investigate the microstructure of polycrystalline silicon (px-Si) thin films which are deposited directly onto glass substrates using reactive magnetron sputtering in a single-pumpdown, two step growth process. px-Si films are of technological interest as thin film transistors in flat panel displays and as absorber layers in solar cells. In the first step, we deposit a microcrystalline Si (µmc-Si:H) film  400 Å can easily be obtained. We will also report preliminary electrical characterizations in both the as-deposited and post-hydrogenated states.

## Thin Films Division
## Room 310 - Session TF-ThA

### Ex-situ Characterization of Thin Films
**Moderator:** P. Ruzakowski Athey, PPG Industries Inc.

2:00pm **TF-ThA1 Ex-situ Characterization of Polycrystalline Thin Films**, *K. Barmak,* ***J. Rickman,*** Lehigh University **INVITED**

The granular nature of polycrystalline thin films is an increasingly important consideration in modern high technology applications as grain and structural dimensions become comparable. This realization has prompted a renewed interest in understanding those factors which affect the evolution of grain structure in both as-deposited and reacted thin films. This presentation will address the ex-situ characterization of thin films that are of relevance to microelectronics interconnects and magnetic storage media. Experimental evidence from differential scanning calorimetry, x-ray diffraction and transmission electron microscopy studies will be reviewed and details of transformation kinetics and grain structure evolution will be discussed. Theoretical models and computer simulations of these processes will also be presented. Finally, results of algorithm development for automated grain size measurement from transmission electron micrographs will be highlighted.

2:40pm **TF-ThA3 TEM Study of Defects, Domains and Vacancy Ordering in Ga@sub 2@Se@sub 3@/GaAs(100) and Ga@sub 2@Se@sub 3@/Si(111) Thin Films**, ***Z.R. Dai***, *S.R. Chegwidden, S. Meng,* University of Washington*; K. Ueno, A. Koma,* University of Tokyo, Japan*; M.A. Olmstead, F.S. Ohuchi,* University of Washington

Ga@sub2@Se@sub3@ belongs to a class of M2(III)X3(VI) compounds (where M=AL, Ga or In and X=S, Se or Te) that is largely unexplored, but which has potential applications in novel optoelectronic devices. The crystal structure of Ga@sub2@Se@sub3@ is based on tetrahedral atomic coordination as in the zinc-blende structure, but in which one third of Ga sites on the average are left as vacancies to preserve charge balance in the crystal. The existence of vacancies complicates the Ga@sub2@Se@sub3@ crystal structure, with their arrangement leading to a variety of polymorphs and possible defect structures, but the arrangement of vacancies also strongly influences the properties of Ga@sub2@Se@sub3@. In particular, the vacancy ordering in the crystal structure likely results in the unique optical and electronic properties of Ga@sub2@Se@sub3@. In this work, the Ga@sub2@Se@sub3@ films were grown on GaAs(100) and Si(111) substrates, respectively, by Molecular Beam Epitaxy (MBE). The microstructure of the Ga@sub2@Se@sub3@ films were characterized by transmission electron microscopy (TEM). For the case of Ga@sub2@Se@sub3@/GaAs(100), a single crystal Ga@sub2@Se@sub3@ film was obtained, in which a number of plane boundaries parallel to [111] crystal planes were observed, as well as some micro-twins. The electron diffraction analysis indicates that the vacancies are ordered to distribute into the domains separated by the boundaries in some equivalent orientations of the zinc-blende structure. For the case of Ga@sub2@Se@sub3@/Si(111), the Ga@sub2@Se@sub3@ film consists of domains forming a twin relationship each other, relative to the [-110] crystal planes of the Si substrate. The vacancy ordering occurs in the [111] crystal planes and the periodicity of atomic stacking along crystal direction is two times of that for the cubic zinc-blende structure. The ordered arrangement of the vacancies is associated with the surface structure of the substrates. The attribute of the boundaries and formation mechanism of the domains and vacancy ordering will be discussed.

3:00pm **TF-ThA4 Sputtered Deposition of Ni@sub 3@Al Thin Films**, *G.B. Thompson, X.D. Zhang, R. Grylls,* ***R. Banerjee****, P.M. Anderson, H.L. Fraser,* Ohio State University

Monolithic Ni@sub 3@Al films have been deposited using a magnetron sputtering technique. Each film was deposited onto an amorphous SiO@sub 2@ substrate at 25°C (unheated), 200°C, and 400°C. X-ray Diffraction, Transmission Electron Microscopy, and High Resolution Electron Microscopy was used to characterize the microstructure of the thin films. A nonequilibrium structure, which can be common in PVD techniques due to the high quench rates from a vapor to solid state, was observed. The phase transition towards the equilibrium state was seen with increased substrate temperature. X-ray diffraction indicated a strong [111] texture to all the deposited films in the growth direction. Although the elevated temperature samples showed the onset of a slight cubic orientation texturing. TEM/HREM was performed upon cross section and plan view specimens. An equiaxed grain structure, with a typical grain size of 20 nm, was seen in the films. No obvious macroscopic grain growth was observed for the elevated temperature samples. However a high density of planar defects was observed in the cross section of the unheated specimen. Formation of these planer defects will be discussed in terms of phase transition and structural stability in the Ni@sub 3@Al thin films.

3:20pm **TF-ThA5 Roughness Measurements With X-Rays Using an Out-Of-Plane Scattering Geometry**, ***J.J. Kelly IV***, *J. Con Foo, J.F. MacKay, M.G. Lagally,* University of Wisconsin, Madison

Surface and interface morphology plays a dominant role in fields ranging from thin-film magnetism to integrated-circuit production. Of the several techniques that can be used routinely for quantitative determination of the surface morphology, diffuse X-ray scattering provides the widest dynamic range in terms of the lateral scale of roughness that can be sampled, from sub-Angstrom to many micrometers. The use of a grazing-incidence geometry enhances surface sensitivity. In investigations to assess the influence of surface and interface roughness in giant-magnetoresistive films, we have measured the diffusely scattered intensity of soft X-rays (~1 keV) from rough surfaces and interfaces of Si, Co, Ni, and their combinations, using a non-conventional approach, an out-of-plane scattering geometry. An out-of-plane measurement is not limited by the shadow edge of the sample surface, and thus it samples a much wider range of reciprocal space and hence a much larger range of roughness wavelengths than a conventional in-plane rocking curve. We use the Born approximation and the distorted wave Born approximation to fit the data. The bandpass of our experiment allows us to extract all the relevant roughness parameters: the mean square roughness @sigma@, the correlation length @xi@, and the roughness exponent h. We have measured a roughness of bare highest-quality polished silicon wafers of less than 0.5 @Ao@, lower than can be measured by AFM or other techniques. Comparisons to rocking curves measured on the same samples in the same chamber will be presented to show the capabilities of this method. Roughness measurements on epitaxial sputter-deposited Co-on-Cu films will also be shown and, as time permits, compared to magnetic properties of these films. This work is supported by AFOSR and NSF. The Synchrotron Radiation Center is supported by the NSF.

3:40pm **TF-ThA6 Accurate Thin Film Density Measured by Energy-Dispersive X-ray Reflectivity**, ***W.E. Wallace***, *W.L. Wu,* National Institute of Standards and Technology

Recent advances in our group in x-ray reflectivity have shown that this technique, applied in the proper fashion, can be a robust and rapid way to measure thin film density on flat substrates. Density is directly related to most thin film properties: dielectric constant, moisture absorption, thermal diffusivity, etc. Until now, measuring the mass density of thin films less than a 1000 nm thick has not been an easy task. Energy-dispersive X-ray reflectivity,@footnote 1@ performed on a modified x-ray powder diffractometer, has been applied to a variety of organic and inorganic thin film materials. The density can be measured to ±1% in a few minutes. Changes in density of a variety of materials have been followed as a function of processing conditions, for example, annealing time and temperature of spin-on-glasses. A description of the technique and some recent representative examples of low dielectric constant materials for ULSI will be given. @FootnoteText@ @footnote 1@W.E. Wallace and W.L. Wu, Applied Physics Letters 67(1995)1203

4:00pm **TF-ThA7 Structure and Electronic Properties of the Novel Semiconductor Alloy Cd@sub 1-x@Cu@sub x@Te**, *S. López-López, G. Torres-Delgado,* ***S.J. Jiménez-Sandoval****, O. Jiménez-Sandoval, R. Castanedo-Pérez,* Cinvestav-IPN, Mexico

In this work is presented an investigation of the structural and electronic properties of the novel semiconductor alloy Cd@sub 1-x@Cu@sub x@Te. The samples were prepared as thin films by rf sputtering on substrates made of Corning glass. X-ray diffraction patterns showed that the incorporation of Cu into CdTe did not affect significantly its lattice parameter. The band gap of the alloys measured by optical transmission spectroscopy was smaller than that of pure CdTe by only 50 meV, approximately. Due to its sensitivity to local atomic order, the samples were studied by micro-Raman spectroscopy. The transverse and longitudinal optic modes regularly observed in CdTe were also found in the Cd@sub 1-x@Cu@sub x@Te samples with frequency variations lower than 2 cm@super -1@, and no additional modes were observed. From the Raman experiments it was also determined that the incorporation of Cu precluded the formation of Te aggregates, which are commonly detected in CdTe thin films and bulk samples. The Cd@sub 1-x@Cu@sub x@Te films

were p-type and, remarkably, it was found in some cases resistivity reductions by seven orders of magnitude with respect to pure CdTe films grown under the same conditions. This is opposite to the observed behavior on Cu-doped CdTe samples where copper acts detrimentally for electronic transport. Our studies indicated that the samples with the best properties were those in which the copper content was below ca. 7.0 at%.

4:20pm **TF-ThA8 Thickness-Dependence of Infrared Reflection-Absorption Spectra from Thin Film of Anatase-type TiO@sub2@ Grown on Polished MgO(001) Substrate by Ar-ion Beam Sputtering**, *D. Osabe, T. Uchitani,* ***K. Maki,*** Yokohama City University, Japan

The structure of thin film of TiO@sub2@ grown on air-cleaved surface of MgO(001) by Ar-ion beam sputtering on a Ti target is controlled by adjusting the partial pressure of O@sub2@ vapour (P@subO@) flowing near the substrate, which has previously been published in J.Vac.Sci. Technol., A, 15, 2485(1997). In the present study we will show that the preferred oriented anatase-type thin film of TiO@sub2@ with its c-axis parallel to the surface is grown on polished MgO(001) substrate held at 550 ˚C in P@subO@ = 1.1 x 10@super-2@ Pa. The polished substrate was used for studying optical property after X-rays diffraction. The diffraction peak height from the (200) lattice plane of the anatase TiO@sub 2@ film increases sigmoidally with increasing its thickness, d, below 20 nm, and linearly with d above 20 nm. The relationship between infrared reflection-absorption peak height near at 510 cm@super-1@ and d also shows the similar one between the diffraction peak height and d. Some discussion is given how to determine the dielectric function, @epsilon@, as a function of the angular frequency, @omega@, from the infrared reflection-absorption(IR-RAS). @epsilon@(@omega@) is determined by comparing the measured IR-RAS with the calculated one which is performed by evaluating from the Fresnel coefficient for the anisotropic materials on isotropic substrate by adopting the Lorentz model for the dielectric constant. After determining the dielectric function, the ratio of @omega@ for exciting of the longitudinal optical phonon, @omega@@subL@, to the transverse one, @omega@@subT@, that is, so called Lyddane-Sachs-Teller relation, is determined, which equals to the square root of @epsilon@(0)/@epsilon@. For this aim IR-RAS at 100 @<=@ @omega@ @<=@ 1000 cm@super-1@ is required which is shown for rutile-type single crystal of TiO@sub2@. However, IR-RAS at 400 @<=@ @omega@ @<=@ 1000 cm@super-1@ in the present study will be available for judging whether epitaxially-grown thin film is prepared or not and will show that the dielectric property of crystalline regions in thin films of TiO@sub2@ at d > 20 nm are similar to the bulk single crystal.

4:40pm **TF-ThA9 Rutherford Backscattering and Channeling Studies of Al and Mg Diffusion in Iron Oxide Thin Films**, ***S. Thevuthasan,*** *W. Jiang, D.E. McCready, S.A. Chambers,* Pacific Northwest National Laboratory*; N.R. Shivaparan, R.J. Smith,* Montana State University

There is growing interest in the epitaxial growth of model oxides on various oxide and metal substrates to obtain high-quality surfaces and films. This interest is being fueled by the utility of these materials in magnetic recording, surface geochemistry, heterogeneous catalysis and integrated microwave devices. A number of single crystal iron oxide films with various stoichiometries and orientations have recently been synthesized in our lab using oxygen-plasma-assisted molecular beam epitaxial growth. In the present work, we have used Rutherford backscattering and channeling techniques to investigate the crystalline quality of epitaxially grown @alpha@-Fe@sub 2@O@sub 3@(0001) on Al@sub 2@O@sub 3@(0001), Fe@sub 3@O@sub 4@(001) on MgO(001), and @gamma@-Fe@sub 2@O@sub 3@(001) on MgO(001). The nature of the film-substrate interface, the crystallographic quality of the films, and Al, Mg, and Fe interdiffusion were investigated. The minimum backscattering yields obtained from channeling and random spectra show that in general the film crystal quality is reasonably good. However, @alpha@-Fe@sub 2@O@sub 3@(0001) grown on Al@sub 2@O@sub 3@(0001) show some disordering at the interface due to the 5.7% lattice mismatch. In contrast, no disorder was seen at the Fe@sub 3@O@sub 4@(001)/MgO(001), and @gamma@-Fe@sub 2@O@sub 3@(001)/MgO(001) interfaces, in keeping with their respective lattice mismatches of -0.36% and -0.89%. Mg appears to outdiffuse into @gamma@-Fe@sub 2@O@sub 3@ film at a lower temperature than that at which Al outdiffusion occurs into epitaxial @alpha@-Fe@sub 2@O@sub 3@. Interestingly, Fe indiffusion was not observed for the @gamma@-Fe@sub 2@O@sub 3@/MgO system until higher temperatures (~800°C) were reached. @FootnoteText@ Work supported by the U.S. Department of Energy, Offices of Basic Energy Sciences and Biological and Environmental Research - Environmental Management Science Program and NSF Grant DMR-9409205

5:00pm **TF-ThA10 Advances in the Characterization of Thin (<30 nm) TiN Films Using SIMS**, ***A.V. Li-Fatou,*** *G.R. Mount, V.K.F. Chia,* Charles Evans & Associates

Titanium and titanium nitride films are widely used as barrier stacks to prevent junction spiking. It is also an important material for anti-reflection coatings (ARCs) on aluminum films to facilitate lithography processes during multilevel metallization for the manufacture of integrated circuits on silicon-based semiconductor devices. SIMS (secondary ion mass spectrometry) is a very capable tool for characterizing films because of its excellent detection sensitivities for transition elements and atmospherics. However, as films become thinner (<30 nm) quantitative analysis by SIMS also becomes more challenging. This is because a larger fraction of the film is now located in the transient region of the depth profile where the ion yields are not yet constant. In this paper we describe the effects of various analytical conditions (primary beam energies, incidence angles, and oxygen flooding using quandrupole and magnetic sector mass spectrometers) on the sputter rate at the near-surface and ion yields at the film/substrate interface. The samples used in this study were CVD grown samples of Ti (10 nm) /Si and Ti (3x10 nm)/Si. Our preliminary study shows that oblique angle bombardment with oxygen flooding can result in both accurate quantification and depth calibration in the upper 15 nm of the sample; there appears to be a dependence between the incidence angle and the primary beam energy. Interfacial mixing is reduced by using a lower primary beam energy. Ion yield enhancements are reduced using oblique incidence bombardment and oxygen flooding. Difficulties still exist when using SIMS to determine the exact the film thickness.

## Thin Films Division
## Room 310 - Session TF-FrM

### Thin Film Deposition from Chemical Precursors

**Moderator:** A. Belkind, Stevens Institute of Technology

8:20am **TF-FrM1 Annealing of Copper Electrodeposits**, ***C.H. Seah***, *S. Mridha,* Nanyang Technological University, Republic of Singapore; *L.H. Chan,* Chartered Semiconductor Manufacturing Ltd., Republic of Singapore

Cu is the best metallization candidate to replace Al and its alloys because of its lower resistivity and better electromigration resistance. Significant progress has been made in building multilevel Cu interconnection systems for advanced microelectronics. As a result, it is important to understand how the morphology of the electroplated Cu films and its properties change after annealing at high temperatures. The properties of annealed electroplated Cu films, together with the diffusion barrier performance, have been studied. Electroplating of Cu films was performed onto p-Si with Cu and W seed layers. The barriers film are either TiN, Ta or TaN. The specimens were then annealed at 600 and 700°C in N@sub 2@ atmosphere for grain growth study. The average grain size of the as-plated Cu films was found to be different; larger Cu grains were formed on W seed layer compared to that formed on Cu (600 vs 200 nm). After annealing, all the Cu films recrystallized readily and grain growth occurred. Regardless of the initial grain size of the electroplated Cu films, the final grain size after annealing was found to be similar in both seed materials. The grain sizes were about 1.0 and 1.2 µm after annealing at 600 and 700°C respectively. The driving force for grain growth is the surface energy release from elimination of grain boundaries and thus achieving an equilibrium state. The annealed films produced a layered microstructure, together with the presence of pinholes and cavities. Results show that all the diffusion barriers remained intact after annealing at 600°C. The CVD TiN barrier appeared to fail at 700°C annealing if the film stack contains only 100 Å thick barrier layer. The resistivity of the electroplated Cu films was also found to be reduced from 2.1 microhm-cm to 1.9 microhm-cm after annealing at both temperatures.

8:40am **TF-FrM2 Polymerized C-Si Films on Metal Substrates: Cu Adhesion/Diffusion Barriers for ULSI?**, *L. Chen,* ***J.A. Kelber,*** University of North Texas

The increasing demands of ULSI have created a need for Cu diffusion/adhesion barriers which are consistently effective at thicknesses of several hundred angstroms or less. Further, the integration of Cu with low-dielectric polymers requires the reliable adhesion at the Cu/polymer interface. We have recently synthesized polymeric C-Si films which are resistant to thermal diffusion of Cu at temperatures below 800 K, even at thicknesses less than 100 Å. These films also show potential for providing a covalently bonded interphase between Cu and, e.g., fluoropolymer dielectrics. The films are created by electron or UV photon bombardment of condensed vinyl silane derivatives. The films are not SiC, and have chemical compositions very close to those of the vinyl silane precursors (e.g., Si:C = 1:4 for vinyltrimethylsilane(VTMS)-derived films). TPD data show that polymerization occurs via the vinyl group. Films derived from VTMS are adherent and stable on Ta substrates until 1000 K in UHV. Diffusion of Cu is not observed below 800 K. Cu adhesion to the substrate is relatively good, with dewetting of deposited overlayers occuring only at temperatures above 600 K in UHV. Perfluorobenzene moieties can also be incorporated into the growing film with good thermal stability, indicating that these films have potential to adhesively couple Cu surfaces to vapor-deposited fluoropolymer films. Opportunities for molecular tailoring of electronic and mechanical properties via systematic variation of precursor structure will be discussed.

9:00am **TF-FrM3 Ultra High Rate, Wide Area, Plasma Polymerized Films from High Molecular Weight/Low Vapor Pressure Liquid or Solid Monomer Precursors**, ***J.D. Affinito***, *M.E. Gross, P.A. Mounier, S. Stockhause,* Pacific Northwest National Laboratory **INVITED**

A new process has been developed for the high rate vacuum deposition of solid films from high molecular weight/low vapor pressure liquid, or even solid, monomer precursors. The gas resulting from the flash evaporation of a liquid monomer mixture, or from a suspension of liquid monomer and insoluble solid particles, is used as the support medium for a glow discharge in a Plasma Enhanced Chemical Vapor Deposition-like (PECVD) process. Due to the high molecular weight/low vapor pressure nature of the precursors, the plasma of the flash evaporated gas cryo-condenses at extremely high rate on substrates at ambient, and higher, temperatures. Upon condensation the liquefied plasma immediately begins to polymerize to form a solid film due to the high concentration of radicals and ions contained in the liquid film. The process has been successfully implemented in a vacuum roll coating system in a roll-to-roll deposition process. Polymer films, and Molecularly Doped Polymer (MDP) composite films of polymer and light emitting organic molecules, have been deposited at thickness' ranging from 0.1 microns to 24 microns at webs speeds as high as 100 linear meters per minute. This new deposition process will be discussed along with some properties of the films fabricated with this new process.

9:40am **TF-FrM5 Effects of Temperature (350 - 25°C) on OH Incorporation and Electrical Performance of Plasma Deposited Silicon Dioxide Thin Films for Applications on Plastic Substrates**, ***A. Gupta***, *C.G. Makosiej, G.N. Parsons,* North Carolina State University

Very low temperature PECVD SiO@sub 2@ may be useful on plastic substrates for gate dielectrics for polysilicon thin film transisitors, or for barrier or insulating layers in organic LED displays. We have used SiH@sub 4@/N@sub 2@O/He/H@sub 2@ mixtures in a parallel plate rf plasma CVD reactor to form SiO@sub 2@ on silicon, and examined the effect of substrate temperature between 350°C and 25°C, rf power, and gas ratios on the chemical composition, refractive index, etch rate, chemical stability, deposition rate, and I-V and C-V characteristics. Low temperature films have also been deposited on PET and polycarbonate substrates to examine adhesion and stress. Films deposited at 350°C show good insulating properties with breakdown fields >8 MV/cm. As temperature is decreased to 100°C, an increase in charge trapping is observed. The decrease in temperature also results in an increase in deposition rate from 65 to 100 Å/min, and a decrease in refractive index from 1.47 to 1.43, indicating a less dense structure. Infrared spectroscopy shows a correlation between OH concentration and the Si-O stretch peak position. At 90W, as the temperature decreases from 350 to 250°C, the Si-O stretch mode position decreases from 1063 to 1056 cm@super -1@, indicating an increase in bond strain. As temperature is decreased from 250 to 25°C, the Si-O peak increases from 1056 to 1065 cm@super -1@, due to an increase in OH incorporation during deposition. After deposition, the OH concentration increases with ambient exposure, and decreases slightly during annealing at 300°C. Process variations, including H@sub 2@ and He dilution, and time modulated deposition and H@sub 2@ exposure cycles to decrease OH incorporation have been tested. Films with less OH in the as deposited condition show a greater resistance to post-deposition oxidation, and show improved electrical properties. Films with good leakage characteristics (

10:00am **TF-FrM6 Structural and Electrical Properties of SrTiO@sub 3@ Thin Films Prepared by Plasma Enhanced MOCVD**, *D.O. Kim,* ***Y-.B. Hahn***, Chonbuk National University, Korea

Dielectric SrTiO@sub 3@ ultra thin films having 30 - 75 nm thickness were deposited on Pt/Si and Ir/Si substrates by plasma enhanced MOCVD using high purity Ti(O-i-C@sub 3@H@sub 7@)@sub 4@, Sr(tmhd)@sub 2@ and O@sub 2@. The structural and electrical properties of SrTiO@sub 3@ films were studied in terms of crystallinity, microstructure, current leakage, and dielectric constant. For the case of Pt/Si substrate, the peaks of (100) and (111) SrTiO@sub 3@ together with TiO2 and SrCO@sub 3@ peaks started to appear at 500 @super o@C, and at 550 @super o@C for (110) and (211) peaks without TiO@sub 2@ and SrCO@sub 3@ peaks. For Ir/Si substrate, peaks of (100) SrTiO@sub 3@ and TiO@sub 2@ appeared at 500 @super o@C, and at 550 @super o@C for (110), (111) and (210) SrTiO@sub 3@. Dielectric constants decreased as the film thickness decreased. The leakage current density of the SrTiO@sub 3@ films decreased with increasing deposition temperature up to 550 @super o@C, but somewhat increased at > 550 @super o@C. It was found that the electron current was limited by tunneling effect for films thinner than 30 nm, but limited by Schottky emission for films thicker than 30 nm. The electron affinity and depletion layer of SrTiO@sub 3@ films, based on the electron current mechanism, were 4.0-4.3 eV and 15 nm, respectively.

10:20am **TF-FrM7 Thermal Stability of MOCVD TiN/PECVD SiOF Interface for Cu Metallization**, ***K.H. Kim***, *S.J. Park, G.S. Lee,* Louisiana State University

RC delay based on Al and SiO@sub 2@ interconnection system comprises a great portion of the total delay as circuit density increases. RC delay can be reduced by using low resistance metal or low dielectric constant insulator, or both. Thus, Cu and SiOF are preferred to be used as low resistance and low dielectric materials, respectively. However, Cu metallization system needs a stable diffusion barrier. Meanwhile, it is known that the properties of MOCVD TiN film are affected largely by the substrate material. In this

study, we investigated the interface stability of MOCVD TiN/PECVD SiOF film for the possibility of integration. The SiOF film of 100 nm thickness was deposited using TEFS with Ar as a carrier and O@sub 2@ as a reactant. The MOCVD TiN film of 50 nm thickness was coated using TDEAT and He carrier at substrate temperature of 350°C. The TiN/SiOF film was annealed in the temperature range of 200 - 600°C for 30 min. in vacuum. The effect of annealing was investigated by FTIR, four point probe, C-V measurement, and AES. It was observed that the sheet resistance of the TiN film increased and the dielectric constant of the SiOF film decreased at annealing temperature above 500°C. After the TiN film was removed from the annealed TiN/SiOF film, FTIR showed unknown peak which increased as a function of annealing temperature. The results of C-V measurement also showed unstable interface at annealing temperature above 500°C. We demonstrated that the properties of MOCVD TiN/PECVD SiOF film were affected by annealing, which may be due to diffusion and interaction at interface.

10:40am **TF-FrM8 Assessment of As-deposited Polycrystalline Silicon Films on Polymer Substrates using ECR-PECVD***, **S.H. Bae**, S.J. Fonash,* The Pennsylvania State University

As-deposited polycrystalline silicon (poly-Si) films have been successfully grown on polymer substrates at 120 and 200 °C using ECR (Electron Cyclotron Resonance) PECVD. PES (polyethersulfone) and PET (polyethylene terephthalate) substrates have been used in this work. To block diffusion and degassing from the polymer substrates, 1000 Å ECR-PECVD silicon nitrides (refractive index = 1.9) have been coated on the substrates prior to Si film depositions. Then, Si films have been deposited with SiH@sub 4@/H@sub 2@ ECR plasmas. The structural, opto-electric and electrical characteristics of Si films have been assessed by X-ray diffraction, photoluminescence, and electrical conductivity measurements, respectively. X-ray diffractions for 3500 Å Si films grown at 120 and 200 °C have shown (111), (220), (311) and (331) peaks which are the primary diffraction peaks of crystalline Si materials. We have explored the effect of the 13.56 MHz RF substrate bias during growth of Si films. According to X-ray diffraction patterns, low RF substrate bias and high substrate temperature are favorable for high degree of crystallinity of Si films. It is noted that RF substrate bias during ECR plasmas may tailor electrical properties of as-deposited poly-Si films; as the RF substrate bias is applied and increased, conductivities are improved and activation energies are decreased. In photoluminescence spectra of Si films, the intensity of luminescence is enhanced as RF bias is increased. Since intensity of photoluminescence is related to defect density of Si films; the more defects offer the more alternative paths for non-radiative recombination which compete with radiative recombination paths, enhancement of photoluminescence intensity with applying RF bias may mean that degree of defect passivation during Si film growth is controlled by RF substrate bias.

11:00am **TF-FrM9 A Novel Method For Determining Kinetic Rate Expressions For CVD Processes Using a Combination of Step Coverage Measurements and Computer Simulation***, E.J. McInerney, **G. Ramanath**, D.C. Smith,* Novellus Systems

There is an increasing need for understanding reaction kinetics and mechanisms during chemical vapor deposition (CVD) to accurately predict deposition rate, uniformity, step coverage, and reactor throughput. We present a novel method for determining reliable kinetic rate expressions by a combination of transport modeling and experimental measurements of step coverage and planar deposition rate. We demonstrate the method by applying it to W CVD during H@sub 2@ reduction of WF@sub 6@ to elucidate -- for the first time -- two distinct kinetic mechanisms operating in different WF@sub 6@ flow regimes. Rate expressions deduced from deposition rate measurements on planar geometries often fail to accurately predict step coverage due to incorrect assumptions of near-surface concentration N@sub s@ of reactants. As direct measurements are difficult, modeling species transport in the reactor is necessary to estimate N@sub s@. However, this method by itself is inadequate because small errors in the model can lead to large errors in the rate expression. In our method, we obviate the limitations of traditional modeling by utilizing experimentally measured step coverage values to model species transport within topological features in addition to that in the reactor. In this way N@sub s@ is calculated self-consistently, allowing the determination of the reaction order n and kinetic mechanisms. Despite numerous studies of the H@sub 2@-WF@sub 6@ reaction over the last 30 years, the order of WF@sub 6@ concentration dependence on W deposition rate has been unresolved between n=0, 1/6, and 1. Our results reveal two distinct mechanisms: at high WF@sub 6@ flows the rate is HF desorption limited (n=1/6), while at low flows WF@sub 6@ adsorption is the limiting step (n=1).

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