Monday Morning, November 3, 2003

Thin Films
Room 329 - Session TF-MoM

Atomic Layer Deposition

Moderator: S.M. Rossnagel, IBM T.J. Watson Research Center

8:40am **TF-MoM2 Advancements in ALD Chemistry***, **M. Ritala**,* University of Helsinki, Finland **INVITED**

The atomic layer deposition (ALD) method has gained continuously increasing interest as a future manufacturing technology for microelectronics, and also several other potential application areas have been pointed out. This interest arises from the excellent conformality and large area uniformity and the atomic level composition and thickness control offered by ALD through its self-limiting film growth mechanism. While the ALD method is currently widely examined worldwide, a majority of the research is still largely focused on rather limited number of processes. A wider application of ALD in microelectronics and elsewhere is directly dependent on the availability of appropriate processes. There is thus a continuous need for developing cost-effective ALD processes for materials of an interest, like metals, metal nitrides, and high-k and ferroelectric oxides. In this presentation, recent advancements in ALD process development will be presented, mostly focusing on metals. Challenges for the future research will be discussed too.

9:20am **TF-MoM4 Incorporation and Control of Silica in Hafnium Silicates using Atomic Layer Deposition Techniques***, **M.N. Rocklein**, F.H. Fabreguette, S.M. George,* University of Colorado

HfO@sub 2@ and hafnium silicates are leading high-k candidates for replacing SiO@sub 2@ as the gate dielectric in MOSFETs. Advantages of incorporating silica in HfO@sub 2@ are to reduce the interfacial oxide and to prevent crystallization. Very few studies have reported incorporation of silica in HfO@sub 2@ by atomic layer deposition (ALD). We have investigated a new silicon precursor for hafnium silicate ALD. Although sequential exposures of this Si-precursor and H@sub 2@O will not lead to continuous SiO@sub 2@ ALD, growth of hafnium silicates is possible by mixing this Si-precursor and H@sub 2@O exposures together with HfO@sub 2@ ALD from 200 - 260 °C. HfO@sub 2@ ALD is conducted using tetrakis-diethylamidohafnium (TDEAH). Compositional control of the hafnium silicate Hf@sub x@Si@sub (1-x)@O@sub 2@ is achieved by controlling the relative number of Si-precursor/H@sub 2@O and TDEAH/H@sub 2@O reaction cycles. Silica incorporation and control is demonstrated using quartz crystal microbalance (QCM), transmission Fourier tranform infrared (FTIR) spectroscopy and x-ray photoelectron spectroscopy (XPS) investigations. Various reactant exposure sequences yield SiO@sub 2@ content ranging from 0 - 70 %. Hafnium silicate growth rates are also measured and confirmed using a variety of techniques including QCM, spectroscopic ellipsometry, and x-ray reflectance (XRR). Growth rates decrease with increasing SiO@sub 2@ content and vary from 0.65 Å/cycle at 21% SiO@sub 2@ to 0.18 Å/cycle at 67% SiO@sub 2@.

9:40am **TF-MoM5 In-situ Infrared Analysis of Atomic Layer Deposition Half-Reactions: Hafnium Diethyl- Amide Adsorption and Dissociation on SiO@sub2@ and HfO@sub2@***, **M.J. Kelly**, T.D. Abatemarco, G.N. Parsons,* North Carolina State University

A critical challenge in ALD is to control bond structure at the initial heterostructure interface. For example, the deposition of metal oxide dielectric materials on silicon results in unwanted substrate oxidation and uncontrolled interface structure. To better understand elementary ALD half reactions, we have built an in-situ attenuated total internal reflection infrared spectrometry system, and used it to observe adsorption, desorption and decomposition of Hf diethlyamide [Hf(N(CH@sub 2@CH@sub 3@)@sub 2@)@sub 4@] (HDA) on oxidized silicon and on deposited HfO@sub 2@ at substrate temperatures from 50 to 300°C. The effect of water exposure on adsorbed HDA was also characterized under some conditions. We find that for adsorption on thin HfO@sub 2@ at 50 and 175°C, the C-H stretching modes indicate adsorption and saturation of the molecular precursor at typical dosing pressures (10-40mTorr), followed by desorption upon pumping, consistent with molecular physisorption at low temperature. At 300°C, minimal CH stretching mode adsorption is observed, consistent with rapid decomposition of the precursor upon adsorption. At intermediate temperatures (250°C), non-self-limiting adsorption and decomposition are observed, and Si-O-H bond formation indicates silicon oxidation. Analyzing the precursor uptake curves as a function of temperature, we find that the rate of adsorption on HfO@sub 2@ is thermally activated with an effective barrier of ~0.1eV, consistent with a reduction in entropy upon adsorption. Results will be discussed in terms of the inter-relation of precursor structure and surface pretreatment on the energetics and kinetics of precursor reaction and substrate oxidation.

10:00am **TF-MoM6 Characteristics of Hafnium Aluminate Films for Gate Dielectric Applications Deposited by Atomic Layer Deposition Method***, **J. Lee**, J. Koo,* Hanyang University, Korea*; Y. Kim,* Pusan National University, Korea*; H. Jeon,* Hanyang University, Korea

The decrease of SiO@sub 2@ gate oxide thickness in sub-100nm metal-oxide-semiconductor (MOS) technology causes high leakage current and serious device reliability problems. To avoid these problems, high-k materials such as HfO@sub 2@ and Al@sub 2@O@sub 3@ have been mainly investigated as a potential candidate to substitute SiO@sub 2@ gate dielectric. However, these high-k materials have some supplement to be solved for applying to MOS device. Hafnium aluminate films would solve relatively high leakage current and low dielectric constant problems of HfO@sub 2@ and Al@sub 2@O@sub 3@, respectively. Hafnium aluminate films were expected to have amorphous structure of Al@sub 2@O@sub 3@ while maintaining high dielectric constant of HfO@sub 2@. Hafnium aluminate films were deposited on p-type Si substrates by atomic layer deposition (ALD) method using HfCl@sub 4@ and trimethylaluminium (TMA) as Hf and Al precursors, respectively, with H@sub 2@O as reaction gas. All samples were rapid thermal annealed at 800°C for 10 seconds in N@sub 2@ ambient. Forming gas anneal was performed in an H@sub 2@+N@sub 2@ atmosphere at 450°C for 30 minutes after Pt gate electrode deposition. The physical and chemical properties were analyzed by cross sectional transmission electron microscope (XTEM), Auger electron spectroscopy (AES) and x-ray photoelectron spectroscopy (XPS). The electrical and reliability characteristics including equivalent oxide thickness (EOT), hysteresis, leakage current and interface defects were analyzed by I-V and C-V measurements. In this study, the characteristics of hafnium aluminate films for gate dielectric applications deposited by atomic layer deposition method were investigated. The chemical compositions and heat-treatments effects on the electrical and reliability characteristics of hafnium aluminate films for gate dielectric applications will be mainly presented. @FootnoteText@ @footnote 1@M. Leskela, M. Ritala, Thin Solid Films, 409(2002), 138, 146.

10:20am **TF-MoM7 Atomic Layer Deposition of Transition Metal Films***, **R.G. Gordon**,* Harvard University **INVITED**

Effective reactions have been discovered for ALD of many metal compounds, including oxides, nitrides, sulfides and fluorides. However, ALD of pure metals has proven to be more difficult. Tungsten and ruthenium are the only metals previously deposited from molecular precursors by self-limiting, complementary ALD reactions. ALD of titanium, tantalum, copper and aluminum used atomic hydrogen as a reactant, which limits the step coverage attainable in deep trenches because of the rapid recombination of hydrogen atoms. It is also difficult to design ALD apparatus to distribute hydrogen atoms over large areas, and to avoid plasma damage to substrates. We synthesized many new metal acetamidinates, a class of metal precursors that we designed to have the properties needed for ALD of metals. These compounds have the required high thermal stability because two metal-nitrogen bonds hold each ligand onto the metal (chelate stabilization). Nevertheless, each metal-nitrogen bond is individually weak (formally a bond order of only one half) and highly reactive. The high volatility of these metal compounds (vaporization temperatures less than 100 C) arises from the outer surfaces of the molecules being entirely saturated hydrocarbons. For example, homoleptic N,N'-diisopropylacetamidinato metal compounds and molecular hydrogen gas were used as ALD reactants to form highly uniform and conformal thin films of transition metals including iron, cobalt, nickel and copper. We propose that these ALD layers grow by a catalytic hydrogenation mechanism that should also operate during the ALD of many other metals. The process should allow improved production of many devices, such as copper interconnections in microelectronics, magnetic information storage, micro-electromechanical structures and catalysts. Use of water vapor in place of hydrogen gives highly uniform, conformal films of metal oxides, while ammonia gives metal nitrides.

11:00am **TF-MoM9 Demonstration of AlN ALD Using Hydrazine as the Nitrogen Precursor***, **F.H. Fabreguette**,* University of Colorado*; T.E. Seidel,* Genus, Inc.*; S.M. George,* University of Colorado

Aluminum nitride (AlN) has attracted widespread attention due to its remarkable thermal, electronic and mechanical properties leading to

numerous applications. AlN growth by chemical vapor deposition (CVD) is accomplished using trimethylaluminum (TMA) and ammonia (NH@sub 3@) precursors. AlN atomic layer deposition (ALD) has also been attempted using TMA and NH@sub 3@. Unfortunately, high ALD temperatures are required where TMA pyrolysis is a problem. Lower ALD reaction temperatures where TMA will not decompose can be obtained using hydrazine, N@sub 2@H@sub 4@ , as the nitrogen precursor. This study presents the use of hydrazine as a new route for AlN ALD at temperatures as low as 177°C. AlN ALD using both ammonia and hydrazine was monitored and compared using an in-situ quartz crystal microbalance (QCM). ALD uptake curves showed 'soft' saturation behavior for the TMA half-reaction and 'hard' saturation for the hydrazine half-reaction. The AlN growth rate using hydrazine was found to be about 1.0 Å/cycle at 177°C. The ammonia reactant produced a growth rate < 0.1 Å/cycle. Different characterizations were performed on the AlN ALD films grown at 177°C. Ellipsometry and x-ray reflectivity confirmed a growth rate of about 1 Å/cycle. The AlN ALD films were determined to be amorphous by x-ray diffraction and smooth when measured by atomic force microscopy. X-ray photoelectron spectroscopy showed aluminum and nitrogen in the films and an oxide surface layer. Electrical measurements for the dielectric constant and the leakage current density were in good agreement with results from AlN CVD films. This study demonstrates the potential of hydrazine as a suitable nitrogen precursor for low temperature AlN ALD.

11:20am **TF-MoM10 Low Temperature Deposition of SiN using Sequential Exposures of Si@sub 2@Cl@sub 6@ and NH@sub 3@**, *J.Y. Ahn, J.G. Kim,* ***J.W. Lim****, H.S. Kim, U. Chung, J.T. Moon,* Samsung Electronics, Korea

Low temperature deposition of SiN is required in many applications for fabrication of high performance MOSFET devices. SiN films were deposited by atomic layer deposition (ALD) and cyclic chemical vapor deposition (CVD) using sequential exposures of Si@sub 2@Cl@sub 6@ and NH@sub 3@ at temperatures ranging from 400 to 600°C. The films were deposited in a mini-batch reactor employing cross-wafer gas flow scheme for rapid delivery of precursors and rapid evacuation of reactants. The saturated growth rate of ALD-SiN was 2.2Å/cycle. The films were characterized by AES, AFM, FTIR, RBS, SIMS, SEM, spectroscopic ellipsometry and TEM. The wet etch and reactive ion etch (RIE) characteristics were also investigated. ALD-SiN films are smooth, highly conformal and stoichiometric. In addition, they contain less hydrogen than the conventional LPCVD-SiN films deposited at equivalent temperatures, and exhibit high etch selectivity to SiO@sub 2@. The RIE selectivity to SiO@sub 2@ was determined to be nearly constant within the temperature range of 400-600°C. The properties of the films deposited by cyclic CVD method, in which self-limited surface saturation is not obtained, were comparable to ALD-SiN, and the Si:N ratio of cyclic CVD-SiN is controllable in the range of 0.7 - 1.0 by varying the Si source exposure and dilution. The electrical properties of the ALD and cyclic CVD films were evaluated by performing I-V and C-V measurements with MIS (Metal Insulator Silicon) capacitor structures. The physical and electrical properties of cyclic CVD films deposited at 590°C were as good as the conventional LPCVD films deposited at 700°C using DCS/NH@sub 3@ chemistry, and therefore the thermal budget for device fabrication can be reduced using cyclic CVD-SiN. The suitability of ALD-SiN films deposited at 450°C was reviewed for fabrication of high performance CMOS devices employing NiSi module process.

11:40am **TF-MoM11 Atomic Layer Deposition on Fine Particles**, *J.D. Ferguson, A.W. Weimer,* ***S.M. George****,* University of Colorado

Because of the self-limiting nature of the sequential surface reactions in atomic layer deposition (ALD), atomic layer controlled and conformal films can be deposited on fine particles. Our recent work has investigated coatings on BaTiO@sub 3@ particles that are used to fabricate multilayer capacitors. SiO@sub 2@ has been coated on BaTiO@sub 3@ particles using NH@sub 3@-catalyzed SiO@sub 2@ ALD with tetraethoxysilane (TEOS) and H@sub 2@O as the reactants. ZnO has been coated on BaTiO@sub 3@ particles using ZnO ALD with diethylzinc (DEZ) and H@sub 2@O as the reactants. The ALD surface chemistry has been investigated using Fourier transform infrared (FTIR) spectroscopy. The FTIR studies observe the exchange of surface species as expected from the surface chemistry. In addition, the growth of the bulk SiO@sub 2@ and ZnO films versus number of reactant cycles is also observed in the FTIR spectra. Further confirmation of the SiO@sub 2@ ALD and ZnO ALD is obtained from transmission electron microscopy (TEM) analysis of the BaTiO@sub 3@ particles. The TEM analysis reveals conformal SiO@sub 2@ films with a SiO@sub 2@ growth rate of 0.7 Å per TEOS/H@sub 2@O reaction cycle at 300 K. TEM measurements also observe conformal ZnO films with a ZnO growth rate of 2.2 Å per DEZ/H@sub 2@O reaction cycle at 450 K. Additional FTIR and TEM studies have explored Al@sub 2@O@sub 3@ ALD on polyethylene (PE) particles at 350 K using trimethylaluminum (TMA) and H@sub 2@O as the reactants. The deposition of inorganic oxides on polymers may be important as gas diffusion barriers for packaging applications. The FTIR investigations observe the exchange of surface species during Al@sub 2@O@sub 3@ ALD and the growth of Al@sub 2@O@sub 3@ bulk features. The TEM measurements reveal a conformal Al@sub 2@O@sub 3@ film on the surface of the PE particles. ALD on large quantities of fine particles will be demonstrated using fluidized particle bed ALD reactors.

Thin Films
Room 329 - Session TF-MoA
Atomic Layer Deposition and Low-k

Moderator: S.M. George, University of Colorado

2:00pm **TF-MoA1 The Application of Plasma for Metal Atomic Layer Deposition for Cu Interconnect Technology***, S.M. Rossnagel, **H. Kim,*** IBM T.J. Watson Research Center **INVITED**

As the semiconductor dimensions scale down to sub-65 nm regime with implementation of Cu interconnect technology, the need for introducing metal thin film deposition techniques with excellent conformality and thickness control at the nanometer scale has been increased. Atomic layer deposition (ALD) is expected to play an important role in depositing thin layers in nanoscale Si device manufacturing. Among the key materials used for modern semiconductor processing, thin films of inert, refractory or noble materials will continue to be used in interconnect applications as diffusion barriers, seed and adhesion layers as well as potential front end applications such as contacts or gate metallization. The plasma technologies that have been widely used for various aspects of the semiconductor device processing can be extended into ALD areas. Plasma enhanced ALD (PE-ALD) allows deposition at significantly lower temperatures with better film properties than both conventional thermal ALD and chemical vapor deposition. This low temperature process makes PE-ALD more attractive for emerging low-k interconnect materials. In addition, since ALD is surface-sensitive deposition technique, surface modification by plasma exposure can be used to alter nucleation and adhesion. In this presentation, we will present the PE-ALD of Ta-based thin films for Cu interconnect technology and the surface modification by plasma for metal and nitride ALD processes.

2:40pm **TF-MoA3 Copper Atomic Layer Deposition Using In Situ-Generated Cu@sub 3@Cl@sub 3@ and Hydrogen Radicals***, **M.D. Groner**, S.M. George,* University of Colorado

Copper (Cu) atomic layer deposition (ALD) has been achieved using in situ-generated Cu@sub3@Cl@sub3@ and hydrogen radicals. The Cu ALD chemistry involves copper chloride adsorption on the substrate followed by copper chloride reduction to copper metal using hydrogen radicals. The copper chloride precursor, Cu@sub3@Cl@sub3@, is generated in situ when a small Cl@sub2@ or SnCl@sub4@ gas pulse transported by a carrier gas contacts a high surface area Cu braid at T> 200 °C. The in-situ generation of Cu@sub3@Cl@sub3@ overcomes the stability problems associated with using a heated CuCl source to generate the copper chloride. The Cu ALD is performed using a "moving sample" reactor in which a pneumatic transfer arm shuttles the sample between the Cu@sub3@Cl@sub3@ dosing region and the inductively coupled plasma (ICP) hydrogen radical source. This reactor design is important because close proximity to the hydrogen radical source is necessary to overcome efficient hydrogen radical recombination on the reactor walls. Cu ALD films have been grown on glass, sapphire, and silicon substrates. The Cu ALD films nucleate well on the various substrates after hydrogen radical exposures. Cu ALD growth rates of >1 Å/cycle have been achieved as determined by real-time in situ optical thickness measurements of the Cu film growth. The Cu films are very shiny and copper-colored and display a high reflectivity. X-ray diffraction (XRD) analysis of the Cu films show distinct (111), (200), (220) and (311) diffraction peaks with no additional XRD peaks. Preliminary studies have shown successful Cu ALD at substrate temperatures ranging from ~200-260 °C. Further results will be presented on the dependence of growth temperature, copper chloride precursor exposure and hydrogen radical exposure.

3:00pm **TF-MoA4 Dynamic Equipment and Process Simulation for Atomic Layer Deposition Technology***, **W. Lei,** Y. Cai, L. Henn-Lecordier, G.W. Rubloff,* University of Maryland

While the self-limiting growth of atomic layer deposition (ALD) makes it a promising process for thin film deposition, the rapid precursor gas cycling required poses manufacturability challenges. We have developed dynamic simulation models for equipment and process behavior to explore the influence of detailed design on : (1) surface chemistry and resulting material quality; (2) manufacturing throughput; (3) materials utilization for environmental (ESH) and cost metrics; and (4) real-time sensor system design for integrated metrology and advanced process control. The dynamic simulators are flexible in accommodating a variety of process and surface chemistries as well as equipment designs. For a mini-chamber design motivated by manufacturing throughput requirements, we have compared operational modes based on static vs. dynamic gas flow, which shows ESH benefits for the former without sacrifice of throughput. The simulator reveals time-dependence analysis of ALD dynamics as a function of surface chemistry and equipment design, providing guidance for integrated metrology. Based on these results, we are developing a novel ALD reactor for process and metrology development which emulates the conditions found in emerging commercial reactors. @FootnoteText@ Supported by the National Institute of Science and Technology and by the NSF/SRC Center for Environmentally Benign Semiconductor Manufacturing.

3:20pm **TF-MoA5 Al@sub 2@O@sub 3@ Atomic Layer Deposition for the Enhancement of MEMS Performance and Reliability***, **C.F. Herrmann**, N.D. Hoivik, F.W. DelRio, V.M. Bright, Y.C. Lee, S.M. George,* University of Colorado

Ultrathin and conformal films deposited using Atomic Layer Deposition (ALD) can enhance the reliability and performance of MEMS devices. Al@sub 2@O@sub 3@ ALD films are particularly useful because the Al@sub 2@O@sub 3@ ALD surface chemistry is very favorable and amenable to growth on a wide variety of substrates. Al@sub 2@O@sub 3@ ALD films on electrostatically-actuated polysilicon cantilever switches were found to prevent electrical shorting and increase the number of actuation cycles before device failure. In addition, the resonant frequencies of the cantilever beams were also increased by Al@sub 2@O@sub 3@ ALD films resulting from added stiffness. ALD can also be utilized to deposit robust and reliable hydrophobic coatings. The initial MEMS device is optimized for hydrophobic precursor attachment by: 1) covering the MEMS surface uniformly with a continuous adhesion layer; 2) providing a high surface coverage of hydroxyl groups for maximum precursor attachment; and 3) smoothing and removing nanometer-sized capillaries that may otherwise lead to microcapillaries and stiction problems. Quartz crystal microbalance (QCM) studies have been used to monitor the attachment of various chlorosilane and dialkylaminosilane hydrophobic precursors on the Al@sub 2@O@sub 3@ ALD adhesion layer. The QCM results are consistent with the deposition of a dense hydrophobic film on the Al@sub 2@O@sub 3@ adhesion layer. The hydrophobic films on Al@sub 2@O@sub 3@ ALD adhesion layers on silicon wafers were observed to increase dramatically the contact angle. The film properties and adhesion energies were also measured for hydrophobic films on MEMS cantilever beams. After submersion in water, the hydrophobic-coated beams showed much less stiction than the uncoated beams.

3:40pm **TF-MoA6 Processes and Properties of Porous CVD Low-k Materials***, **Y. Travaly**, M. Van Hove, G. Beyer,* IMEC / SPDT / ITSMI, Belgium*; K. Maex,* IMEC / SPDT, Belgium **INVITED**

The aggressive down scaling of device dimensions in integrated circuits requires the introduction of non-traditional materials such as porous CVD low-k dielectrics together with thinner copper diffusion barrier with improved step coverage and conformality along via and trench side walls. This results in a number of concerns with respect to both the low-k film and Cu diffusion barrier to be implemented. Regarding the low-k dielectric, it appeared over the last few years, that not only its electrical properties are of importance but also its compatibility with the process steps encountered in the course of its integration. We therefore identified a number of key properties such as porosity, bulk diffusion of wet chemicals, slurries and barrier precursors, etc. and studied them in relation with surface sealing by plasma treatments, liner deposition or metallic barrier. For the Cu barrier, a number of factors come into play and influence dramatically the interconnect delay (RC delay) especially for the most aggressive pitches. Among these factors, one can easily control the quality of the barrier interface with the low-k or the Cu as well as the minimum barrier thickness. The cross-sectional barrier profile in interconnect appears also to be of prime importance for both RC delay and barrier reliability. To improve the step coverage and minimize the barrier area, we have considered various approaches including a highly conformal atomic layer deposition (ALD) process. However, when used in combination with porous low-k dielectrics, an ALD process gives rise to other issues such as the diffusion and interaction of ALD precursors with the various layers (low-k, hard masks) present in the dielectric stack. We studied these interactions and identified suitable methods to prevent detrimental effects. Ultimately, we studied the reliability of the Cu/low-k system in relation to barrier integrity.

4:20pm **TF-MoA8 Deposition of Low k OSG Films Exhibiting Enhanced Mechanical Properties by PECVD**, *J.L. Vincent, R.N. Vrtis,* ***A.S. Lukas,*** *M.L. O'Neill, B.K. Peterson, M.D. Bitner, G.J. Karwacki,* Air Products and Chemicals, Inc.

Silica is the traditional insulating material used in interlayer dielectrics and has a k of 3.9-4.2. To achieve the lower k's required for the next generation of IC's, methyl (Me) groups may be added to the silicate structure by using organosilane precursors. The lower k in the resulting organosilicate glass (OSG) is attributed to the decreased density, increased hydrophobicity, and reduction of the polarizability of the material. We previously demonstrated the use of diethoxymethylsilane (DEMS) to deposit OSG films by PECVD with k's of 2.7-3.3 (dense) and 2.0-2.5 (porous). However, this decrease in k comes at the expense of the mechanical properties due to the disruption of the silicate network by terminal Si-Me. Our modeling studies show that the benefit of adding Me to lower the k diminishes after a Si/Me ratio of about 2/1 in the OSG material, yet the mechanical properties continue to decline as the Si/Me ratio approaches 1/1. Our dense DEMS OSG films (k = 3.0) are deposited at temperatures > 350 C. These films have excellent mechanical strength and a Si/Me ratio of 2/1. However, our process for porous DEMS OSG (k < 2.5) requires a deposition temperature below 280 C, resulting in a Si/Me ratio close to 1/1 and significantly decreased mechanical strength. We found that the Me incorporation can be reduced in by using a precursor mixture of DEMS and a second silica source without Me groups. This allows the control of the terminal Me content in the final film chemically, as opposed to using process conditions such as high temperature or plasma power. We were successful in depositing OSG films with Si/Me ratios of 2/1 from precursor mixtures at deposition temperatures as low as 200 C. For example, a porous OSG with a post-anneal k of 2.2 and a nanoindention hardness of about 0.6 GPa was deposited at 270 C. This is a nearly 2-fold increase in the mechanical strength as compared to films with identical k deposited without the second silica source.

4:40pm **TF-MoA9 Expanding Thermal Plasma for Low-k Dielectrics: Guiding the Film Chemistry by Means of Selected Dissociation Paths in the Plasma**, ***M. Creatore****, Y. Barrell, W.M.M. Kessels, M.C.M. van de Sanden,* Eindhoven University of Technology, The Netherlands

As the need for low-k dielectrics in the ULSI technology becomes urgent, the research primarily focuses on the deposition of novel materials with appropriate electrical properties and on the challenges concerning their integration with subsequent processing steps. Within this framework, plasma-enhanced chemical vapour deposition of organosilicate glass films is considered promising due their chemical structure similar to SiO@sub 2@, which leads to a simpler process integration. In this contribution we address the remote expanding thermal plasma (ETP) as a novel technique for the deposition of low-k carbon-doped SiO@sub 2@ films from Ar/hexamethyldisiloxane (HMDSO)/oxygen mixtures. We have obtained low-k films which exhibit k values (yet not optimised) in the range 2.9-3.4 (at 1 MHz) and still fairly good mechanical properties (hardness of 1 GPa, modulus of 10 GPa). These results were not expected because literature, in general, reports on low-k films deposited from precursors with 2 Si-O and 1 Si-C bonds per Si atom (e.g., dimethoxymethylsilane and diethoxymethylsilane), in order to reach a compromise between dielectric and mechanical film properties. Our approach, on the contrary, utilizes HMDSO (apparently not suitable due to the high Si-CH@sub 3@: Si-O bond ratio) because the ETP technique allows selecting different fragmentation paths for the precursor and this can eventually be turned to the progressive cleavage of Si-C bonds. The relatively easy control on the process kinetics can be monitored by means of gas phase diagnostics, such as Mass Spectrometry and Cavity Ring Down Spectroscopy. The information on the HMDSO dissociation paths allows tailoring the organic functionalities in the low-k films by means of IR absorption spectroscopy, XPS, and spectroscopic ellipsometry.

5:00pm **TF-MoA10 Photoresist Removal on Porous Low-k Materials Using an Energetic (100s of eV) Oxygen Neutral Beam**, *D.J. Economou,* ***Q. Wang,*** University of Houston*; B. White,* AMD and International SEMATECH*; P.J. Wolf,* Intel and International SEMATECH*; T. Jacobs,* Philips Semiconductors*; J. Fourcher,* International SEMATECH

Integration of porous low-k materials for interconnect technology present many challenges to the etch, ash and cleans processes. One challenge is the post etch removal of photoresist on open porous low-k films. Porous low-k films are very susceptible to damage by plasma processing, which can raise the overall keff of the film. Traditionally, a pure oxygen plasma ash is one method used for photoresist removal on CVD dielectrics. This method cannot be applied to exposed low-k films, because chemical and physical damage occurs. Successful photoresist removal on low-k films can be achieved by reducing chemistries or dilute O2 processes in RIE etch tools. This work shows how an energetic (100s of eV) oxygen neutral beam can be used to strip photoresist, without physical or electrical damage to the exposed low-k material. @FootnoteText@ Work at UH supported by International SEMATECH.

Plasma Science and Technology
Room 315 - Session PS+TF-TuM

Plasma Enchanced Chemical Vapor Deposition
Moderator: D.C. Guerin, Naval Research Laboratory

8:20am **PS+TF-TuM1 PECVD, From the Laboratory to Mass Production***, J.P.M. Schmitt*[1]*,* Unaxis Management Incorporated, Switzerland **INVITED**

After a rapid recall of the "alchemist" age of gas decomposition by an electrical arc, the gradual birth of PECVD is described in the early 70's. The richness of PECVD potential was then realised creating expectations for a long list of potential applications. Soon after the first industrial applications of PECVD were demonstrated. A look back at the pioneering days of PECVD allows to identify and discuss the key attributes of PECVD that made (and keep) this technology attractive for film coating. In the early 80's PECVD was hype and was the objet of active research. Basic research teams activity focused on the complex mechanisms involved in the PECVD process. First were understood the basic steps such as electron induced molecular dissociation and particle-surface processes. It is only later that far more complex mechanisms such as dust formation were found to be also extremely important. The status of knowledge on basic mechanisms will be reviewed. In the last 15-20 years a wide variety of configurations for the plasma reactors were tested. Instead of a complete zoological classification of all variations, we relate various classes of plasmas with their most marking attributes to the PECVD process key mechanisms. If in the 80's PECVD was already at work in mass production plants, it is in the 90's that a full set of production related problems were actively addressed. Self-cleaning was found a highly desirable ability for a PECVD tool. This requirement combined with high throughput demand led to new classes of equipment and processes. The application field of PECVD also stretched from the food industry the most sophisticated high tech industry. PECVD is today facing new challenges. The glass substrate size for the flat display industry is about to exceed 2m@super 2@ still requiring good uniformity and high throughput. PECVD has also the opportunity to prove itself into new fields such as semiconductor epitaxy or deposition of organic based films with functional groups.

9:00am **PS+TF-TuM3 Plasma-enhanced Deposition of Silicon and Metal Oxynitride Films in a High-density Ammonia Discharge***, Z.G. Xiao,* ***T.D. Mantei,*** University of Cincinnati

Silicon, titanium, zirconium, and chromium oxynitride films have been grown in a high-density electron cyclotron resonance (ECR) ammonia discharge. The organosilicon deposition precursors for silicon oxynitride were hexamethyldisiloxane and tetramethylsilane, while the organometallic deposition precursors for metal oxynitride were titanium (IV) isopropoxide and tetrakis(dimethethlyamino)titanium, zirconium 2-methyl-2-butoxide and zirconium t-butoxide, and bis(ethylbenzene)chromium. The plasma-grown films had nanoindentation hardness values of 12 - 14 GPa for SiN, 20 - 28 GPa for TiN, 17 - 21 GPa for ZrN, and 25 - 31 GPa for CrN. Deposition growth rates were 40 - 50 nm/min for silicon oxynitride and 10 - 20 nm/min for the metal oxynitrides. X-ray photoelectron spectroscopic (XPS) analyses showed the nitrogen content of silicon, titanium, and zirconium oxynitrides to be 31% - 38%, while the CrN nitrogen content was 15%. The SiN films grown from hexamethyldisiloxane were colorless and transparent while films grown from tetramethylsilane had the characteristic dark color of Si@sub 3@N@sub 4@. The TiN and ZrN films had the characteristic brass and white gold colors of TiN and ZrN reference samples while the CrN samples were gray. The SiN films lasted 800 hours in an ASTM B117 accelerated salt-fog corrosion test without visible corrosion, and the TiN and ZrN films lasted 1000 hours without visible color change or corrosion.

9:20am **PS+TF-TuM4 Identification of the Growth Precursors for Hydrogenated Amorphous Carbon Growth***,* ***J. Benedikt***[2]*, R.V. Woen, M.C.M. van de Sanden,* Eindhoven University of Technology, The Netherlands

The plasma chemistry and plasma composition of argon/acetylene expanding thermal plasma, used for fast (up to 70 nm/s) hydrogenated amorphous carbon (a-C:H) film deposition, was studied by means of Cavity Ring Down absorption Spectroscopy and Mass Spectrometry. Since the electron temperature in expanding thermal plasma beam is low (less than 0.3 eV) electron impact processes can be neglected and acetylene dissociation is argon ion induced. The C@sub 2@H radical was identified as the main growth precursor for hard (14 GPa) diamond-like a-C:H films. It was shown that under conditions where most of acetylene molecules are decomposed into C, CH and C@sub 2@ radicals, the a-C:H films are soft and polymer-like. Furthermore it was observed that good a-C:H films can be grown also under conditions when C@sub 4@H@sub 2@ molecules are the main plasma chemistry product and when reactive radicals as C@sub 2@H has already reacted away with acetylene in the gas phase. The mass spectrometry measurements of C@sub 4@H@sub 2@ molecule in the background of the plasma shows that C@sub 4@H@sub 2@ density depends on the wall condition (argon plasma activated or hydrogen plasma passivated) suggesting that C@sub 4@H@sub 2@ is one of the contributors to the film growth. C@sub 2@H and C@sub 4@H@sub 2@ as possible precursors for a-C:H growth is confirmed by recent Molecular Dynamics simulations which reveal reaction probabilities close to one for both species.

9:40am **PS+TF-TuM5 Characterization of TaN Diffusion Barrier Layers Prepared by Chemical-Enhanced Physical Vapor Deposition (CEPVD)***,* ***N. Li***[3]*, D.N. Ruzic,* University of Illinois, Urbana-Champaign

CEPVD of TaN is a novel process attempting to deposit diffusion barrier layers with both high conformal step coverage (as in CVD) and superior quality (as in PVD). The experiments are performed by sputtering a Ta target in a modified conventional PVD instrument and simultaneously adding a certain amount of chemical precursor, TBTDET, in the vicinity of the substrate at elevated temperature (330@degree@C) in combination with a carrier gas (N2), reducing agent (H2), non-reactive sputtering gas (Ar) and a RF-powered secondary ionization plasma. Different combinations of RF power, N2, H2, Ar flow and bias voltage result in distinct resistivity regimes. Increasing H2 flow rate from 5 sccm to 10 sccm allows more hydrocarbon formation and thus results in significant resistivity variation. The addition of 10sccm Ar increases target sputtering and more Ta flux, producing film with relatively lower resistivity (5200 Î¼ -cm compared to 62,000 Î¼ -cm). The addition of Ar also produces a more columnar and porous structure. N2 flow rate determines precursor residence time and so controls growth density and deposition rate. Biasing the substrate with -60 V drops resistivity one order of magnitude. Patterned wafers with various trench aspect ratios are lined to compare the step coverage under different processing conditions. Four point probe, SEM, AES, XRD and XPS are utilized to characterize the film properties and the analysis reveals the balance between energetic Ta flux, TBTDET breakup and impurity volatilization. The synergy between PVD and CVD is clearly demonstrated.

10:20am **PS+TF-TuM7 Anisotropic Cu Deposition using Plasma Chemical Vapor Deposition***,* ***M. Shiratani****, K. Takenaka, M. Takeshita, M. Kita, K. Koga, Y. Watanabe,* Kyushu University, Japan **INVITED**

We have demonstrated complete filling of trenches by anisotropic Cu deposition, in which Cu is filled preferentially from the bottom of the trenches, using plasma chemical vapor deposition.@footnote 1@ The key to realize the anisotropic deposition is kinetic energy and flux of ions irradiating on the surface, since the deposition rate increases with increasing the kinetic energy and fluxes. Previously, by using H-assisted plasma CVD we have realized conformal deposition of smooth 20 nm thick Cu films in trenches as well as conformal filling of trenches.@footnote 2@ Although the Cu films have a low as-deposited resistivity of of 1.85 µ@ohm@cm and a strong adhesion strength above 10 MPa to the TiN layer, conformal filling results in a small crystal grain size below half of the trench width and in a seam where impurities of high concentration remain. The anisotropic deposition offers a possibility to overcome such shortcomings for the conformal filling together with two additional interesting features. One is the fact that deposition rate increases with decreasing the width of a trench. The other is a self-limiting deposition by which deposition stops automatically just after filling completely a trench. This feature may realize a LSI fabrication processes without the chemical mechanical polishing, being attractive for the Cu-porous low-k interconnects. @FootnoteText@@footnote 1@ K. Takenaka, et al., Matr. Sci. Smiconductor Processing 5, 301 (2003).@footnote 2@ M. Shiratani, et al., Sci. and Technol. of Adv. Mater. 2, 505 (2001).

[1] **AVS 50th Anniversary Invited Speaker**
[2] **PSTD Coburn-Winters Student Award Finalist**
[3] **PSTD Coburn-Winters Student Award Finalist**

11:00am **PS+TF-TuM9 Pulsed-plasma Deposition of Silicon Dioxide in a High Density Oxygen Discharge*, Y. Qi*,** *T.D. Mantei,* University of Cincinnati

Hard clear silicon dioxide films have been grown from octamethylcyclotetrasiloxane (OMCTS) at low substrate temperatures in a pulse-modulated high density electron cyclotron resonance (ECR) oxygen plasma. The input microwave power at 2.45 GHz was pulse-modulated with repetition frequencies from 20 Hz to 20 kHz, duty ratios (on-time/period) from 5% to 100%, and peak microwave power levels from 800 W to 2400 W. The resulting films were SiO@sub 2@-like with Si-O bonds and Si:O ratios close to 1:2. The deposition growth rates were almost independent of frequency for all pulse repetition frequencies from 20 Hz to 20 kHz. The growth rates increased strongly as the peak pulse power was increased; with a 50% duty ratio, the growth rate was 0.5 - 0.6 mm/min with 800 W peak power, increasing to 0.8 - 0.9 mm/min at 1600 W peak power. The coating hardness values decreased with pulsed operation as the average input microwave power decreased. Deposition substrate temperatures were significantly lowered as the duty ratio (and thus the average power) decreased, e.g., substrate temperatures were 140°C - 150°C after 10 minutes of deposition with 1600 W of continuous microwave power, dropping to 90°C with a 50% pulse duty ratio and 1600 W peak power. Results from current experiments on pulsed low temperature growth of metal nitride and teflon coatings will also be discussed.

11:40am **PS+TF-TuM11 Secondary Plasma Based Debris Mitigation for Next-Generation 13.5nm EUVL Sources*, B. Jurczyk*,** *D.N. Ruzic, E. Vargas-Lopez, M. Neumann, M. Williams, C. Chrpbak, S. Taj,* University of Illinois at Urbana-Champaign

Next-generation EUV photolithography machines (>25kW-class) require order of magnitude improvements in debris removal for component lifetime and stable operation. Discharge plasma light sources, such as the dense plasma focus, are leading candidates for EUV. The Illinois Debris-mitigation Experiment and Applications Laboratory (IDEAL) consists of a dense plasma focus discharge source operating on order of 25 J/pulse, 100 Hz rep rate, and 4 kV. Argon and Helium gases have been tested to generate plasma environmental conditions similar to that experienced by industry. The secondary-plasma-based debris mitigation technique is presented; a concept pioneered from iPVD reactors at the University of Illinois. Sputtered electrode and chamber component debris is re-ionized in the secondary plasma region and removed with the application of electric fields prior to the collection optics. A helical resonator inductive coil generates the secondary plasma with minimal coil self-biasing for decrease erosion. A dual-channel foil trap, with independently biased plates (0-1kV), collects debris from the secondary plasma region. The foil trap is positioned to vary aspect ratios from 1:1-to-16:1 to correlate with gas pressure effects. Results from in-situ high-precision quartz-crystal-oscillators, ex-situ surface characterization (XPS, Auger, Profilometry, etc.), secondary plasma characterization, and collection optic protection factors are presented for a series of mitigation schemes.

Thin Films
Room 329 - Session TF-TuM

Thin Films on Organic, Polymeric and Biological Substrates

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

8:20am **TF-TuM1 Low Cost Thin Film Electronics on Flexible Polymeric Substrates*, T.N. Jackson,*** Penn State University **INVITED**

Silicon microelectronics has been spectacularly successful at providing increasingly complex digital processing and large-capacity digital memory with continually improving performance/$. It has been less successful at providing simple function at very low cost and in providing electronic function over large areas or on arbitrary surfaces. Thin film transistors (TFTs), based on either inorganic or organic semiconductors, are of interest for lowcost flexible and arbitrary substrate applications. Inorganic amorphous silicon (a-Si:H) TFTs are widely used as pixel access devices in displays and large area sensors on glass substrates. It is also possible to fabricate these devices on polymeric or other unconventional substrates and we have demonstrated a-Si:H TFTs on high temperature polymeric substrates (for example, polyimide) with performance very similar to that obtained on glass substrates. Organic semiconductors are of particular interest because they can be deposited and processed at very low temperature (often less than 100°C). Organic thin film transistor (OTFT) device performance now rivals or exceeds that of a-Si:H devices, and low OTFT process temperatures allow fabrication on a range of surfaces including cloth, paper, or polymeric substrates. Using small molecule organic semiconductors we have fabricated TFTs with field effect mobility > 1 cm2/V-sec on flexible polymeric substrates with good uniformity and yield. Devices of either type (organic or inorganic) are of particular interest for applications where their switch characteristics can be used for selection or isolation of arrays of devices. This approach, widely used for display addressing, allows many thousands or even millions of sensor, actuator, or other elements to be controlled with simple, low-cost electronics and the flexible processing used for thin film active devices allows direct integration with a wide range of materials and devices.

9:00am **TF-TuM3 Ultra-high Barrier Coating Deposition on Polycarbonate Substrates*, M. Schaepkens,*** *K.W. Flanagan, T.-W. Kim, M. Yan, A.G. Erlat, C. Heller, P.A. McConnelee,* General Electric

The use of polycarbonate film substrates enables fabrication of new applications, such as flexible display devices, lighting devices, and other flexible electro-optical devices, using low cost, roll-to-roll fabrication technologies. One of the limitations of bare polycarbonate material in these applications is that oxygen and moisture rapidly diffuse through the material and subsequently degrade the electro-optical devices. This paper summarizes recent results obtained at GE Global Research to solve the oxygen and moisture diffusion issue. It will be shown that through the application of thin, dense, plasma-based inorganic coatings one can significantly reduce the oxygen and moisture permeation rate through polycarbonate films. However, as a result of defects that are commonly present in these inorganic coatings there is a limit to the performance of such barrier coatings. To further improve the barrier performance, advanced barrier coatings comprising both inorganic and organic materials have been developed. Both modelling and experimental results will be presented that explain why these hybrid material barrier coatings are capable of reaching ultra-high barrier performance.

9:20am **TF-TuM4 Organic Thin Film Transistors based on Fluorene-Arylene Copolymers for Printed Electronics*, M.G. Dibbs*,** *P. Townsend, D. Brennan, S. Kisting, J. Shaw,* The Dow Chemical Company **INVITED**

During the last 50 years polymeric materials have played an important role in semiconductor fabrication, interconnect, packaging and displays. They have usually played a passive role and have not been involved in the active generation, amplification, and switching of current or voltage. This is now changing. Research efforts on polyanalines, conjugated thiophene oligomers and polymers and pentacene and thiophene oligimers have led to improvements of mobility by 5 orders of magnitude over the last 15 years. Polymeric semiconductors offer a means to create unique, low cost electronic devices since they can be readily fabricated into thin, mechanically robust films onto a variety of substrates by low cost processing techniques. Recently new generations of polymeric semiconductors have been developed. All plastic chips based entirely on organic materials have been demonstrated. Materials based on fluorene-arylene copolymers are under development for this purpose. This paper describes recent work with this family of materials, compares results obtained by different research groups, and discusses issues related to the interplay of device construction and material characterization with a focus toward printed electronics.

10:00am **TF-TuM6 Elastic Metal Interconnects*, J.E. Jones*,** *S.P. Lacour, S. Wagner, Z. Suo,* Princeton University

Stretchable, elastic metal interconnects are a key to the fabrication of 3-D conformal circuits and electrotextiles. The basic concept for reversibly stretchable, elastic metallization is a corrugated stripe of thin-film metal that is expanded by stretching. The maximum elongation is reached when the stripe is stretched flat. We prepared wavy metal stripes by evaporating gold onto pre-stretched strips of the elastomer, poly-dimethyl siloxane (PDMS). We experimented with gold metal line width and thickness and substrate elongation. We measured the film structure, amplitude, and wavelength, as well as electrical resistance in relaxed and various stretched states. So far we have reached elastic strains of 15% while maintaining the initial resistance and 80% with a rise in the resistance. We discovered a rich macroscopic and microscopic film morphology. Presented are the fabrication, electro-mechanical performance, and data on the film structure of these wavy metal interconnects.

10:20am **TF-TuM7 Properties of Indium Zinc Oxide Thin Films on Heat Withstanding Plastic Substrates*, H. Hara*,** *T. Hanada, T. Shiro, T. Yatabe,* Teijin Ltd., Japan

Accompanying with downsizing and improving of resolution of various display devices, a heat withstanding plastic substrates with low resistivity of transparent conductive oxide (TCO) films have been demanded. We

succeeded in development of optical anisotropy transparent plastic substrates with high glass transition temperature over 200 degC. However, tin doped indium oxide (ITO) films used as the transparent conductive electrode have high resistivity about 5E-4 ohm cm because the deposition of ITO films was carried out by sputtering at room temperature to avoid the out gassing from the plastic substrate. This kind of ITO films arise the amorphous/crystal transformation (a/c transformation) at 150 degC. The a/c transformation makes the ITO films crack with increase in resistivity. Then, we have been investigated new transparent conductive oxide films with low resistivity for our heat tolerance plastic substrate. We found that 7.5 wt% ZnO doped indium oxide (IZO(7.5)) films are suitable for the plastic substrates by surveying the ZnO content preciously. IZO(7.5) films deposited at room temperature by conventional dc-magnetron sputtering exhibited the low resistivity, 2.9E-4 ohm cm, keeping the high transparency, over 86 %. By using XRD and TEM, it is revealed that IZO(7.5) films were remained the structure in amorphous after annealing. Moreover, the result of thermal properties of IZO(7.5) films by DSC was elucidated that the a/c transformation was occured at 350 degC. The etching rate to 3.5 wt% HCl of IZO(7.5) films were 2 nm/sec. It is almost as same as or quicker than that of amorphous ITO films. The durability to 5 wt% KOH solution is also good for practical use. We have already succeeded in deposition of IZO(7.5) film on our heat withstanding plastic substrate with 1000 mm width and 500 m length. This substrate with IZO(7.5) film can remain the all properties after 180 degC annealing.

10:40am **TF-TuM8 Interface and Bulk Charge in Low Temperature Silicon Nitride Dielectrics on Plastic Substrates***, **K.J. Park**, G.N. Parsons,* North Carolina State University

Silicon nitride is a common gate dielectric for thin film transistors (TFT's) on plastic substrates, but the effect of processing temperature on charged defects in the film bulk and at the semiconductor/dielectric interface is not well known. For this work silicon nitride was deposited using various NH@sub3@/SiH@sub4@ gas ratios at temperatures between 50° and 300°C and the effect of process conditions on current vs. voltage (IV) and capacitance vs. voltage (CV) measurements was evaluated. For some conditions, CV was measured as a function of film thickness, and values for bulk and interface charge were extracted from the measured trends. We find that the apparent leakage current decreased with increasing NH/SiH ratio, but CV showed that increasing NH/SiH also leads in an increase in the flat band voltage shift, consistent with fixed charge in the films. Thickness dependence of CV indicates that increasing NH/SiH results in an increase in positive fixed charge at the interface, and an increase in negative fixed charge in the film bulk. When the NH/SiH ratio is fixed at 10, changing the substrate temperature from 50 to 250°C results in an increase in positive interface charge, and an increase in negative bulk charge, leading to charge compensation at higher temperatures. Internal charge can lead to a built in field which opposes the applied field, leading to a decrease in leakage current under high internal field conditions. Fixed charge is important to control threshold voltage and may affect transconductance in TFT devices. Results of amorphous silicon TFT's fabricated on polyimide substrates at various temperatures will be discussed.

11:40am **TF-TuM11 Effect of Swift Heavy Ions on the Structural and Optical Properties of RF Plasma Polymerized Aniline Thin Films***, S. Saravanan, C. Joseph Mathai, **M.R. Anantharaman**,* Cochin University of Science and Technology, India*; S. Venkatachalam,* Vikram Sarabhai Space Centre, India*; D.K. Avasthi,* Nuclear Science Centre, India

Organic and polymer thin films have been receiving a great deal of attention due to their interesting properties. They find extensive applications in making devices such as Light Emitting Devices, rechargeable batteries, super capacitors, intermetallic dielectrics and EMI shielding. Polyaniline thin films prepared by plasma polymerization are cross-linked, pinhole free and their permittivity lie in the ultra low k regime. Electronic and photonic applications of polyaniline thin films attracted the attention of various researchers. Modification of polymer thin films by swift heavy ions is well established and ion irradiation of polymers can induce irreversible changes in their structural, electrical and optical properties. Polyaniline thin films prepared by RF plasma polymerization were irradiated with 92 MeV silicon ions for various fluences of 1 x 10@super 11@ ions/cm@super 2@, 1 x 10@super 12@ions/cm@super 2@ and 1 x 10@super 13@ ions/cm@super 2@ using the pelletron facility at Nuclear Science Centre, New Delhi, India. FTIR and UV Vis NIR measurements were carried out on the pristine and silicon ion irradiated polyaniline thin films for structural evaluation and optical bandgap determination. In this paper the effect of swift heavy ions on the structural and optical properties of plasma polymerised aniline thin film is investigated. Their properties are compared with that of the pristine sample. The FTIR spectrum indicates that the structure of the irradiated sample is altered. The optical bandgap of these irradiated thin film is considerably modified.

Thin Films
Room 329 - Session TF-TuA
Transparent Conducting Oxides
Moderator: G. Ockenfuss, OCLI - JDS Uniphase

2:00pm **TF-TuA1 Enhanced Conductivity in Post Deposition Annealed Spinel Oxide Films***, G.J. Exarhos,* Pacific Northwest National Laboratory*;* ***R.R. Owings****,* University of Florida*; C.F. Windisch,* Pacific Northwest National Laboratory*; P.H. Holloway,* University of Florida

Infrared transparent nickel cobalt spinel oxides exhibit p-type polaron conductivity that is highly dependent on the content of nickel in the structure. In this system, the relatively high covalency of nickel and the distribution of cation oxidation states among the available tetrahedral and octahedral lattice sites act to promote polaron formation. The nature of the polaron formed and its mobility are very much dependent upon composition and lattice parameter which, in turn, can be altered by choice of deposition parameters and post deposition treatment. Film conductivity is affected directly by post deposition annealing and can be enhanced or degraded reversibly upon rapid quenching or slow cooling to produce a set of recoverable conductivity values. The addition of lithium into the nickel cobalt system promotes enhanced conductivity as well, if included in small enough amounts. As the nickel concentration in the film increases, less lithium is required to produce a conductivity increase. Lithium containing films also exhibit the same recoverable conductivity behavior when subjected to heat treatment. Electrical property measurements are correlated with vibrational and electron spectroscopy results. Structural nuances are characterized by means of HRTEM. Perturbations to the conductivity are thought to arise from the presence of chemisorbed species such as carbonate and cation partitioning among available sites in the lattice.

2:20pm **TF-TuA2 Reactive-Environment, Hollow Cathode Sputtering: Basic Characteristics and Application to Al@sub 2@O@sub 3@ and Doped ZnO***,* ***A.E. Delahoy****, S.Y. Guo,* Energy Photovoltaics, Inc.

A novel method for thin film deposition by reactive sputtering has been studied. The method is based on metal sputtering in a hollow cathode configuration with supply of a reactive gas to the vicinity of the substrate. The working gas and entrained sputtered atoms exited the cathode through a slot having an aspect ratio of 8:1. The reactive gas is thereby largely prevented from reaching the target. The basic operation of the cathode was studied using a Cu target. These studies included lateral and longitudinal film thickness profiles, the dependence of deposition rate on power, pressure, and flow rate, and film resistivity as a function of substrate temperature and low energy ion bombardment. Al and Zn targets were used to prepare Al@sub 2@O@sub 3@ and ZnO films in a reactive environment of oxygen. Using quartz crystal rate monitoring it was demonstrated that only a very small amount of oxygen passing through the cathode will oxidize (poison) the target, whereas large quantities of oxygen supplied externally to the cathode need not affect the target at all. A very stable plasma discharge and ease of Al@sub 2@O@sub 3@ formation was realized in this latter mode. Using a Zn target, the method was then applied to the preparation of transparent, conductive films of ZnO doped with Al and B. The Al was introduced by co-sputtering, and the B from B@sub 2@H@sub 6@. The dopant concentrations were measured by ICP. The film resistivity was found to depend strongly on oxygen flow rate. Low film resistivities (0.49 x 10@super -3@ ohm-cm) and high deposition rates (a dynamic rate of 17 nm m/min) were achieved.

2:40pm **TF-TuA3 Transparent Electronics: An Overview of Materials, Devices, and Applications***,* ***J.F. Wager****, H.Q. Chiang, D. Hong, B.J. Norris, J.P. Bender, M.M. Valencia, C.-H. Park, J. Anderson, J.Y. Jeong,* Oregon State University*; D.A. Keszler,* Oregon State University, U.S.A*; H. Yanagi, M. Price, J. Tate,* Oregon State University*; R.L. Hoffman,* Hewlett-Packard Company **INVITED**

The objective of transparent electronics is to fabricate invisible electronic circuits. Part of the motivation for the development of transparent electronics is the recent availability of p-type transparent conductive oxides (TCOs). With the emergence of p-type TCOs, in addition to conventional n-type TCOs such as indium-tin oxide, tin oxide, and zinc oxide, fabrication of transparent bipolar electronic devices becomes feasible. This presentation will emphasize four topics. First, a brief overview of conventional n-type TCOs will establish the context for the pursuit of transparent electronics. Second, recent work on the development of p-type TCOs, as well as other non-oxide transparent conductors, will be reviewed. Third, the fabrication and performance of a novel ZnO-based transparent thin-film transistor will be described. Fourth, transparent electronic applications such as transparent select transistors for active-matrix liquid-crystal displays and transparent back-plane electronic drivers for transparent displays will be discussed.

3:20pm **TF-TuA5 Expanding Thermal Plasma Deposition of Textured ZnO: Plasma Processes and Film Growth***,* ***R. Groenen****, I.M. Volintiru, M. Creatore, M.C.M. van de Sanden,* Eindhoven University of Technology, The Netherlands

Zinc oxide (ZnO) is a transparent conducting oxide (TCO) of considerable technological interest. Recently, the utilization of an expanding thermal plasma (ETP) created with a cascaded arc is demonstrated for low temperature textured ZnO deposition.@footnote 1@ The remote configuration of the ETP technique allows for separate control of plasma production, gas phase chemistry and substrate surface treatment and is suitable for large area deposition. Films are deposited on glass substrates from oxygen, diethylzinc and for doped material additionally trimethylaluminum. A controllable, rough surface texture which is essential for application as a front electrode in thin film solar cells, is inherently obtained during deposition. Here, a fundamental study of the plasma processes and film growth is presented. Complementary in-situ diagnostics (e.g. Langmuir probe, quadrupole mass spectrometry, optical emission spectroscopy and spectroscopic ellipsometry) are used to correlate plasma composition, film growth and material properties. Film growth appears to be dominated by molecular (i.e. background) instead of atomic oxygen. The relation between detected species and the observed texture development allows for further improvement of the material light trapping properties. @FootnoteText@@footnote 1@R. Groenen, et al., Appl. Surf. Sci. 173 (2001) 40.

3:40pm **TF-TuA6 Transparent Conducting Amorphous Zn-Sn-O Films Deposited by Simultaneous DC Sputtering***,* ***T. Moriga****, Y. Hayashi, K. Kondo, K. Matsuo, H. Fukumoto, K. Murai, K. Tominaga, I. Nakabayashi,* The University of Tokushima, Japan

The films of ZnO-SnO@sub 2@ system were deposited on glass substrates by simultaneous DC magnetron sputtering apparatus, where ZnO and SnO@sub 2@:Sb (Sb@sub 2@O@sub 5@ 3wt% doped) targets were faced each other. The substrate temparature were maintained at 150 and 250°C. As an experimental parameter, current ratio @delta@, ZnO target current divided by the sum of ZnO and SnO@sub 2@:Sb target currents, was adopted. Monophasic amorphous transparent films appeared for Zn/(Sn+Zn)=0.50-0.73. At Zn/(Sn+Zn)=1/2 (@delta@=0.62), 2/3 (@delta@=0.73) and any other ratio in as-deposited films, neither crystalline ZnSnO@sub 3@ nor Zn@sub 2@SnO@sub 4@ was obtained. Minimum resistivity was found at @delta@=0.50, whose composition was approximately SnO@sub 2@ZnSnO@sub 3@. Amorphous tin oxide coexisting with amorphous zinc stannate ZnSnO@sub 3@ would have an important role to reduce the resistivity. Resistivity increased linearly with an increase of the current ratio, until the composition reached the zinc stannate Zn@sub 2@SnO@sub 4@.

4:20pm **TF-TuA8 Highly Transparent and Conductive ZnO:Al Thin Films Prepared by Vacuum Arc Plasma Evaporation***,* ***T. Miyata****, S. Ida, Y. Minamino, T. Minami,* Kanazawa Institute of Technology, Japan

Recently, we reported preparation of undoped and impurity-doped ZnO thin films on large area substrates by a newly developed vacuum arc plasma evaporation (VAPE) method using oxide fragments as a low-cost source material. Resistivities on the order of 10@super -4@@ohm@cm were obtained in Ga- or F-doped ZnO thin films. However, doping Al into ZnO films was very difficult because of the large difference in decomposition energy (vapor pressure) between Al@sub 2@O@sub 3@ and ZnO. In this paper, we describe the preparation of Al-doped ZnO (AZO) thin films by a newly developed VAPE method using ZnO fragments and a gas source Al dopant. The film depositions were carried out under the following conditions: substrate, large area glass; substrate temperature, RT to 450@super o@C; oxide fragments, sintered ZnO; pressure, 0.08 to 1 Pa; Al dopant gas pressure (flow rate), 0.01 to 0.5 Pa; Ar and O2 gas flow rates, 20 and 0 to 20 sccm; and cathode plasma power, 4.5 to 10 kW. A low resistivity on the order of 10@super -4@@ohm@cm and an average transmittance above 85% in the visible range were obtained in AZO thin films. In addition, a deposition rate of 150 nm/min as well as a uniform distribution of resistivity and thickness on the substrate surface was obtained. It was found that the Al content in AZO films was altered by varying the partial pressure (or flow rate) of the Al dopant gas. It is

concluded that the newly developed VAPE method, using both oxide fragments and gas sources as source materials, is very effective for the preparation of multicomponent oxide thin films.

Wednesday Morning, November 5, 2003

Thin Films
Room 329 - Session TF-WeM
Optical Thin Films and Photovoltaics I

Moderator: R. Sargent, OCLI

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

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

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

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

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

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

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

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

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

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

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

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

Thin Films
Room Hall A-C - Session TF-WeP
Poster Session

TF-WeP1 A Plasma Enhanced Atomic Layer Deposition of Tungsten Nitride Diffusion Barrier for Copper Interconnect, ***H.S. Sim,*** *S.I. Kim,* Korea Institute of Science and Technology*; H. Jeon,* Hanyang University, Korea*; Y.T. Kim,* Korea Institute of Science and Technology, Korea

Tungsten nitride (W-N) was grown on tetraethylorthosilicate (TEOS) by pulse plasma enhanced atomic layer deposition (PPALD) from WF@sub 6@ and NH@sub 3@. It has been very difficult to deposit W-N film on the SiO@sub 2@ surface with ALD method by using WF@sub 6@ and NH@sub 3@ because WF@sub 6@ does not adsorb on the SiO@sub 2@ surface. In this work, however introducing NH@sub 3@ pulse plasma, which modify the SiO@sub 2@ surface to nitride surface, we can deposit the W-N film on the SiO@sub 2@ surface with the growth rate of ~1.3 monolayer/cycle at 350°C. N concentration is also uniformly distributed in the W-N film. This is due to the surface nitridation to enable the adsorption of WF@sub 6@ at the SiO@sub 2@ surface. As a diffusion barrier for the Cu interconnect, electrical measurement reveals that 22 nm thick W-N successfully prevents Cu diffusion after the annealing at 600°C for 30 min.

TF-WeP2 Atomic Layer Deposition of Iron Oxide Thin Films, *T.M. Klein,* ***L.A. Falco,*** University of Alabama

Atomic layer deposition (ALD) has been successfully used in various thin film applications such as electroluminescent (TFEL) flat panel displays, electrochemical solar cells, gas sensors, optical coatings, and microelectronics materials. Thin epitaxial films for super lattices and quantum wells have also been demonstrated by ALD. A wide variety of metal oxide materials have been investigated, however, Fe@sub 2@O@sub 3@ has not been studied extensively. In this poster, we will present the properties of atomic layer deposited hematite (Fe@sub 2@O@sub 3@) thin films on MgO and Al@sub 2@O@sub 3@ substrates using iron (III) acetylacetonate, a solid which sublimes at 110 °C and 2 torr. Iron oxide films may be useful on solid oxide membranes to modify surface selectivity in fuel cell applications.

TF-WeP3 Spatially Regulated Growth of SnO@sub 2@ Thin Films on Si-C Linked Monolayer Template Based on Self-Assembly Technique: Fabrication of Micro Sensor Arrays, ***N. Shirahata,*** *A. Hozumi, Y. Yokogawa, T. Kameyama,* National Institute of Advanced Industrial Science and Technology, Japan*; W.S. Seo,* Advanced Materials Analysis & Evaluation Center, Korea*; K. Koumoto,* Nagoya University, Japan

Increasing attention has recently been paid to the low temperature deposition of SnO@sub 2@ thin films due to its excellent gas sensitivity. The micropatterning of such a conductive material is crucial in order to apply it to microelectronics devices, for example, gas sensor. Unfortunately, due to high chemical reactivity, SnO@sub 2@ is difficult to micropattern with conventional processes using chemical etching. Therefore, the development of an alternative method to fabricate SnO@sub 2@ microstructures having a high resolution is strongly desired. In this study, we report a novel approach to fabricate SnO@sub 2@ micro-array based on the molecular recognition between the precursor molecules and the Si-C linked monolayer. The hydrophobic Si-C linked monolayer substrate was micropatterned by the use of 172 nm vacuum ultraviolet (VUV) light lithography. The VUV irradiated regions became hydrophilic due to the formation of silicon oxide (SiO@sub x@) layer. This micropatterned substrate was immersed into solution containing one of two types of precursors, i.e., SnF@sub 2@ and SnCl@sub 2@Â·2H@sub 2@O (0.03-0.1 M and pH=1.7-5) and kept at less than 80 °C for several hours. After immersion, each sample was sonicated in ethanol in order to remove the physisorbed precursors onto the OD-monolayer (ODM) surface. As confirmed by SEM, AFM and XRD, SnO@sub 2@ thin films with cassiterite structures grew preferentially on the SiO@sub x@ surface while rarely depositing on the ODM surface. As-deposited films consisted of small grains having several tens nm in diameter. Although the crystallinity of the films increased with a decrease of pH values, gas sensitivity of as-formed films to hydrogen molecules was insufficient without annealing. In our case, at least annealing temperature of 300 °C was required in order to attain sufficient gas sensitivity.

TF-WeP4 The Effect of Temperature on the Materials Properties of Low k Films Deposited from Organosilicon Precursors, ***M.L. O'Neill,*** *A.S. Lukas, R.N. Vrtis, J.L. Vincent, E.J. Karwacki, B.K. Peterson, M.D. Bitner,* Air Products and Chemicals, Inc.

Organosilicate glass (OSG) materials are the leading interlayer dielectric (ILD) candidates for current and future technology nodes for integrated circuit manufacture. Current leading edge ILD films are deposited by plasma enhanced chemical vapor deposition (PECVD) with dielectric constants (k) in the range of 2.7-3.2. Future generation ILDs with k < 2.6 require the introduction of porosity. However integration issues caused by the reduced mechanical strength of these materials have delayed their introduction into manufacturing. Although there are numerous ways in which to introduce porosity to a material, the common goal for the processing of these materials is the means to optimize the networking forming process. Herein we examine the effect of temperature on material properties for films produced by PECVD from various OSG precursors. Recently developed codeposition methods use plasma polymerizable organic materials to template porosity in an OSG network during the deposition process. The process requires lower temperature depositions (150-300 °C) to deposit a discrete porogen phase along with the OSG network. Reduced deposition temperatures further compromise material mechanical strength by increasing the number of non-network forming groups in the film. A detailed comparison of film structure and properties with deposition temperature is used to identify the critical components of the precursor in providing the optimal OSG network. Balance between electrical and mechanical properties will provide the maximum opportunity for extension to future generation porous OSG dielectric materials.

TF-WeP5 Photocatalytic Activity and Surface Wettability of TiO@sub 2@/SnO@sub 2@ Heterojunction System, ***N. Kanai,*** *C. Saiki,* The University of Tokyo, Japan*; Y. Fukunaga, M. Abe,* Tokyo University of Science, Japan*; K. Hashimoto, T. Watanabe, H. Ohsaki,* The University of Tokyo, Japan

Photocatalytic efficiency of TiO@sub 2@/SnO@sub 2@ (TiO@sub 2@ overcoated with SnO@sub 2@) heterojunction system was investigated. TiO@sub 2@ and SnO@sub 2@ thin film stacks were deposited on sodalime glass by reactive DC magnetron sputtering using Ti and Sn metal targets. Photocatalytic efficiency of TiO@sub 2@/SnO@sub 2@ thin film stacks was evaluated by gas-phase isopropanol (IPA) photodegradation with UV light irradiation. Photocatalytic efficiency of TiO@sub 2@/SnO@sub 2@ thin film stacks depends on electric conductivity and packing density of overcoating SnO@sub 2@. This result suggests that photogenerated holes might work as a main photocatalysis actor; oxygen defects in SnO@sub 2@ trap photogenerated holes. The results also indicate that the interface between TiO@sub 2@ and SnO@sub 2@ has a great influence on photocatalytic efficiency. Surface wettability of TiO@sub 2@/SnO@sub 2@ thin film stacks was dramatically improved.

TF-WeP6 Characterization of PECVD Low-k Films by Positronium-Annihilation Lifetime Spectroscopy, ***T. Ohdaira,*** *R. Suzuki,* National Institute of Advanced Industrial Science and Technology (AIST), Japan*; Y. Shioya, K. Maeda,* Semiconductor Process Laboratory (SPL), Japan

Positronium-annihilation lifetime spectroscopy (PALS) was used to measure pore size distributions in porous SiOCH films for low-k interlayer dielectrics and Cu-diffusion barriers, which were grown by plasma-enhanced chemical vapor deposition (PECVD) with source gases of HMDSO (hexamethyldisiloxane). In the PECVD, the dielectric constants (k) of the films change with the deposition conditions (pressure, flow rate, RF power, temperature, etc). The PALS analysis showed that the PECVD-grown SiOCH films with k in the range from 2.6 to 4 contain pores with average sizes from 0.4 to 1.2 nm, and that the k values of the films correlate strongly with the pore sizes. The PALS depth profiling was also carried out for the SiOCH low-k films with post plasma treatments and UV irradiations. For the plasma treated films, it is found that the size of the pores in a few 10 nm beneath the surface decreases significantly, indicating that a thin dense layer is formed on the surface.

TF-WeP7 Structural and Electronic Properties of (CdTe)@sub x@(In@sub 2@Te@sub 3@)@sub 1-x@ Thin Films Grown by RF Co-Sputtering@footnote 1@, ***S. Jimenez-Sandoval,*** *M. Melendez-Lira,* Cinvestav-IPN, Mexico*; M. Zapata-Torres,* CICATA-IPN, Mexico

The semiconductor CdTe and In@sub 2@Te@sub 3@ can be employed to form the ternary compound (CdTe)@sub x@(In@sub 2@Te@sub 3@)@sub 1-x@. The compounds CdIn@sub 2@Te@sub 4@ and In@sub 2@Te@sub 3@ have a band gap of 1.12 eV and 1.0 eV respectively. A band gap modulation toward low energies could be achieved by the

incorporation of In in the CdTe lattice. Potential applications for this ternary semiconductor can be thought of in near-IR optoelectronics and photovoltaic heteroestructures. Trying to obtain a gradual incorporation of In in CdTe we produce (CdTe)@sub x@(In@sub 2@Te@sub 3@)@sub 1-x@ thin films by co-sputtering of CdTe and In@sub 2@Te@sub 3@ targets. A previous evaluation of the sputtering yield allow us to control the x parameter through the rf power deliver at each target. We present the results of the chemical, structural and electronic characterization, of a set of eleven thin films, obtained by EDS, X ray diffraction, optical transmission, photoreflectance, photoluminescence, IR and Raman spectroscopies. EDS results indicates that the In content changes between 0 and 38 at%; Cd between 50 and 0 at % and Te between 50 and 62 at %. X ray diffraction showed a gradual change in the CdTe peak positions as In contents increases but for the higher In contents samples were amorphous. Transmission spectroscopy indicates a gradual change of the absorption band edge making difficult to assign the band gap value. Band gap values were obtained by photoreflectance spectroscopy for samples with indium contents up to 17%, indicating a direct band gap. For some samples two transitions were detected. It is possible that for the substrate temperature employed in our system CdTe-rich and In@sub 2@Te@sub 3@-rich compounds were produced with band gap values around 1.43 eV and 1.19 eV, respectively. The results are discussed taking in account the results of the vibrational properties of the thin films. @FootnoteText@@footnote 1@Work partially supported by CONACyT-Mexico.

TF-WeP9 P-Type Semiconducting Cu@sub 2@O-NiO Thin Films Prepared by Magnetron Sputtering, *T. Minami, H. Tanaka,* ***T. Shimakawa,*** Kanazawa Institute of Technology, Japan

This report describes the preparation of p-type semiconducting thin films consisting of a new multicomponent oxide, Cu@sub 2@O-NiO. The Cu@sub 2@O-NiO films were deposited on glass substrates at a temperature of 200-500@super o@C by r.f. magnetron sputtering carried out at a pressure of 0.2-2.0 Pa in an Ar or O@sub 2@ gas atmosphere with an r.f. power of 80 W using a powder target. A mixture of Cu@sub 2@O and NiO powders calcined at 1000@super o@C in an Ar or air atmosphere for 1 h was used as the target: Ni contents (Ni/(Cu+Ni) atomic ratio) in the range from 0 to 100 at.%. The obtained electrical and optical properties of Cu@sub 2@O-NiO thin films were strongly dependent on the deposition conditions as well as the Ni content of the target. P-type semiconducting Cu@sub 2@O-NiO thin films could be prepared at 500@super o@C in a pure Ar gas atmosphere at a pressure of 0.4 Pa using targets calcined in Ar: All the prepared Cu@sub 2@O-NiO thin films were found to exhibit positive hole conduction, as evidenced from Hall measurements and the Seebeck effect. The resistivity of the Cu@sub 2@O-NiO thin films prepared under the above conditions increased markedly as the Ni content was increased up to about 30 at.%, remained relatively constant in the range of about 30 to 60 at.%, and then increased markedly with further increases of Ni content. In the Cu@sub 2@O-NiO thin films prepared with an increasing Ni content in the range from 0 to 30 at.%, the obtained resistivity increased from 70 to 4×10@super 4@@ohm@cm, resulting from decreases in both Hall mobility and hole concentration, and band-gap energy increased from 2.6 to 2.9 eV. In contrast, the Cu@sub 2@O-NiO thin films prepared with a Ni content in the range from 30 to 50 at.% exhibited a relatively constant resistivity on the order of 3×10@super 4@@ohm@cm; these films were identified as the ternary compounds NiCuO@sub 2@ or Ni@sub 2@CuO@sub 3@, by x-ray diffraction analyses. The multicomponent oxide Cu@sub 2@O-NiO thin films prepared by r.f. magnetron sputtering with a Ni content in the range from 0 to 100 at.% were found to be p-type semiconductors with a resistivity that was increased by increasing Ni content. From spectroscopic ellipsometric measurements, it was found that the band-gap energy of Cu@sub 2@O-NiO films can be controlled in the range from 2.6 to 4 eV.

TF-WeP10 Photocatalytic Related Properties and Structure of Titanium Oxide Films, ***C.B. Shiu,*** *M.C. Yang, T.S. Yang,* National Dong Hwa University, Taiwan, ROC*; M.S. Wong,* National Dong Hwa University, Taiwan, ROC, Taiwan, R.O.C

Titanium dioxide (TiO2) films were prepared with ion-assisted electron-beam evaporation technique using rutile TiO2 powder as a source material. Various oxygen flows and ion bombardments were applied to adjust and to modify the composition and the structure of the growing films. Various characterization methods such as ellipsometry, Raman spectroscopy, UV-Visible spectroscopy, XRD, SEM, and TEM were used to measure the crystallinity, morphology, optical and mechanical properties of the films. The photocatalytic related properties under UV and visible lights were characterized by water-contact angle measurement, oxidation of methylene-blue solution, and reduction of Ag from AgCl solution. In general anatase TiO2 films exhibited overall the best photocatalytic performance. However, variations in thickness, composition, phase and crystallinity in the films made a difference in their catalytic behaviors. The effects of film processing parameters on the structure and properties of the oxides will be discussed.

TF-WeP11 Influence of the Microstructure on the Sputter-etching Characteristics of Pulsed-laser Deposited Strontium-titanate-oxide Thin Films, *L. Stafford,* Universite de Montreal, Canada*; M. Gaidi,* INRS-Energie, Canada*; O. Langlois,* Universite de Montreal, Canada*; M. Chaker,* INRS-Energie, Canada*;* ***J. Margot***, Universite de Montreal, Canada*; M. Kulishov,* Adtek Photomask Inc., Canada

Strontium-titanate-oxide (STO) thin films are of great interest for several photonic applications such as high-speed electro-optic elements for next-generation all-optical networks. Obviously, the integration of STO layers into such devices requires a simultaneous optimization of the deposition method and of the patterning process. In this context, we have investigated the influence of the deposition parameters on the etching characteristics. For this purpose, in a first step, stoichiometric SrTiO3 thin films with the crystallographic perovskite structure were grown on a silicon substrate using a Pulsed-Laser Deposition (PLD) technique. The microstructural properties of the films were studied as a function of the buffer gas pressure (O2). In a second step, we have examined the sputter-etching characteristics of the as-deposited films using a high-density argon plasma operated at very low pressure (1 mTorr) and a substrate bias of 100 V. The etch rate of the STO thin films is found to increase with the O2 deposition pressure, a feature that can be related to the microstructural properties of the film (grain size, lattice parameter, etc.) induced by changes in growth conditions. We will show that the etch rate is actually related to the macroscopic film density (film porosity) rather than to its microscopic density (unit cell volume of the film).

TF-WeP12 Ferroelectric Properties of Highly Oriented BLT Films for Ferroelectric-gate Field-effect Transistors, ***J.M. Lee****, C.I. Kim, K.T. Kim,* Chungang University, Korea

Ferroelectric thin films such as Bi-based layered perovskite (SrBi@sub 2@ Ta@sub 2@ O@sub 9@, Bi@sub 4@ Ti@sub 3@ O@sub 12@, Bi@sub 3.25@ La@sub 0.75@ Ti@sub 3@ O@sub 12@, etc.) and Pb(Zr,Ti)O@sub 3@ thin films have been extensively investigated for non-volatile ferroelectric random access memory (FeRAM) devices. Metal-ferroelectric-semiconductor field-effect-transistors (MFSFETs) have advantages of high switching speed, nonvolatility, and high density. However, the MFSFETs have been problem such as interdiffusion between the film and Si. To suppress them, a metal-ferroelectric-insulator-semiconductor (MFIS) structure has been demonstrated. The most important thing in developing a MFIS structure is to find a good insulator that acts as a buffer between the Si substrate and the ferroelectric material, and have relative high dielectric constants, low leakage current, good interface characteristics, and compatibility. The MFIS capacitors were fabricated using a metalorganic decomposition method. Thin layers of Al@sub 2@ O@sub 3@ were deposited as a buffer layer on SiO@sub 2@ /Si and BLT thin films were used as a ferroelectric layer. The electrical and structural properties of the MFIS structure were investigated by varying the Al@sub 2@ O@sub 3@ layer thickness. X-ray diffraction was used to determine the phase of the BLT thin films and the quality of the Al@sub 2@ O@sub 3@ layer. AES and TEM show no interdiffusion that suppressed by using the Al@sub 2@ O@sub 3@ film as buffer layer. The width of the memory window in the C-V curves for the MFIS structure increased with increasing thickness of the Al@sub 2@ O@sub 3@ layer. The experimental results show that the BLT-based MFIS structure is suitable for non-volatile memory FETs with large memory window.

TF-WeP13 The Ferroelectric Properties of Lanthanide-doped Pb(Zr, Ti)O@sub 3@ Thin Films Prepared by using a Sol-gel Method, *C.I. Kim,* ***Y.H. Son****, K.T. Kim,* Chung-Ang University, Korea

The PZT film has a large polarization and a low crystallization temperature. However, there are some problems in the PZT film, such as fatigue with Pt electrodes and poor retention. In this study, lanthanide (Eu, Dy, Tb, Er)-doped lead zirconium titanate(PZT) thin films on the Pt/Ti/SiO@sub 2@/Si substrates prepared by a sol-gel method. According to the ionic radius, lanthanide (Ln) tends to occupy the A-site of PZT perovskite structure and acts as a donor that generates Pb vacancies. Ln doping was found to alter significantly the dielectric and ferroelectric properties. We investigated the effect on the structural and electrical properties of PZT films as a function of Ln concentrations. The structure and the morphology of PZT films were

analyzed by x-ray diffraction (XRD), atomic force microscope (AFM), and scanning electron micrograph (SEM). The depth profile of the interfaces between PZT films and electrode materials were examined using Auger electron spectroscope (AES). SEM and AFM showed uniform surface of PZT films. The dielectric constant and the dielectric loss of Ln-doped PZT thin films decreased with the increasing Ln content. Ln-doped PZT thin films showed improved fatigue characteristic comparing to the undoped PZT thin film.

TF-WeP14 Amorphous Transparent Conductive Oxide Films of In@sub 2@O@sub 3@-ZnO with Additional Al@sub 2@O@sub 3@ Impurities, ***K. Tominaga**, H. Fukumoto, Y. Hayashi, K. Murai, T. Moriga, I. Nakabayashi,* Tokushima University, Japan

In@sub 2@O@sub 3@-ZnO films were deposited by facing target sputtering system of ZnO:Al and In@sub2@O@sub3@ targets. Two targets were sputtered simultaneously in Ar gas at 1 mTorr, and electric current ratio @ganma@=I@sub Zn@/(I@sub Zn@+I@sub In@) was adopted as a deposition parameter to change film composition. Discharge current of each target was changed from 0 to 80 mA in order to change the contents of Zn and In in the film. Compositional ratios of Zn/(Zn+In) in films were nearly equal to @ganna@ value according to the data estimated by X-ray fluorescence analysis. Five ZnO:Al targets containing 0,1,2,3 and 4 wt% Al@sub 2@O@sub 3@ were used for Al adding in the films. In@sub 2@O@sub 3@-ZnO films in amorphous phase were deposited between Zn/Zn+In=0.2-0.6. The lowest resistivity of 1.5x10@super -4@ @ohm@cm was attained in amorphous phase films deposited from In@sub 2@O@sub3@ and ZnO:Al (Al@sub 2@O@sub 3@=2 wt %). Carrier mobility did not depend on Al@sub 2@O@sub 3@ content in ZnO:Al target. Carrier concentrations at the lowest resistivity in amorphous In@sub 2@O@sub 3@-ZnO had the highest value for 2 wt% Al@sub 2@O@sub 3@ content, but decreased at above 3 and 4 wt%. Optical bandgap energy shifted to higher energy side for the film of 4 wt% Al@sub 2@O@sub 3@ content. These results indicate that the energy shift of optical bandgap for amorphous phase film is not ascribed to Burstein-Moss shift. The role of Al in amorphous films is different with that of donors in crystalline In@sub 2@O@sub 3@:Sb and ZnO:Al. On the other hand, Al@sub2@O@sub3@ doping in homologous phase of In@sub 2@O@sub 3@ film decreased carrier concentration, independent on the levels of Al@sub 2@O@sub 3@ doping. This indicates that Al did not act as donor impurities in homologous Zn@sub k@In@sub 2@O@sub k+3@ crystalline films.

TF-WeP15 Luminescence Behavior of Li-doped Gd@sub 2@O@sub 3@ : Eu@super 3+@ Thin Film Phosphors Grown by Pulsed Laser Ablation, ***S.S. Yi,*** Silla University, Korea, South Korea*; J.S. Bae, H.J. Seo, B.K. Moon,* Pukyong National University, Korea*; J.H. Jeong,* Pukyong National University, Korea, South Korea*; P.H. Holloway,* University of Florida

Gd@sub 2@O@sub 3@ : Eu@super 3+@ and Li-doped Gd@sub 2@O@sub 3@ : Eu@super 3+@ luminescent thin films have been grown on Al@sub 2@O@sub 3@ (0001) substrates at substrate temperatures in the range of 500 ~ 700 °C and oxygen pressure in the range of 100 ~ 300 mTorr using a pulsed laser deposition technique. The films grown under different deposition conditions have been characterized using microstructural and luminescent measurements. The photoluminescence brightness data obtained from Li-doped Gd@sub 2@O@sub 3@ : Eu@super 3+@ films grown under optimized conditions have indicated that sapphire is a good substrate for the growth of high quality Li-doped Gd@sub 2@O@sub 3@ : Eu@super 3+@ thin film red phosphor. The luminescence of the Gd@sub 2@O@sub 3@ : Eu@super 3+@ films is highly dependent on the crystallinity and surface roughness of the films. In particular, incorporation of Li@super +@ ions into the Gd@sub 2@O@sub 3@ lattice induces a remarkablely enhanced crystallinity and photoluminescence. The diffraction data suggest that the (222) surface is preferentially oriented parallel to the substrate for films grown on Al@sub 2@O@sub 3@ (0001). In addition, the full width at half maximum (FWHM) of the diffraction peaks is narrower (~20 %) for films grown with versus films grown without Li-doping. The root mean square roughness of these films was found to vary from 7.5 to 16.1 nm depending upon the Li-doping. The highest emission intensity was observed with LiF-doped Gd@sub 1.84@Li@sub 0.08@Eu@sub 0.08@O@sub 3@, whose brightness was increased by a factor of 2.3 in comparison with that of Gd@sub 2@O@sub 3@ : Eu@super 3+@ films. This phosphor is promising for the flat panel displays.

TF-WeP16 Luminescent Characteristics of Se-doped ZnGa@sub 2@O@sub 4@:Mn Thin Film Phosphors Grown by Pulsed Laser Ablation, ***J.H. Jeong,*** Pukyong National University, Korea, South Korea*; J.S. Bae,* Pukyong National University, Korea*; I.W. Kim,* University of Ulsan, Korea*; J.S. Lee,* KyungSung University, Korea*; S.S. Yi,* Silla University, Korea*; P.H. Holloway,* University of Florida

Mn-doped ZnGa@sub 2@O@sub 4-x@Se@sub x@ thin film phosphors have been grown using pulsed laser ablation(PLA) under various growth conditions. The structural characterization was carried out on a series of ZnGa@sub 2@O@sub 4-x@Se@sub x@ : Mn@super 2+@ films grown on MgO(100) substrates using Zn-rich ceramic targets. Zn-rich ceramic targets have been prepared to compensate for the vaporization loss of Zn during PLA. The oxygen pressure was fixed at 100 mTorr and substrate temperatures were varied from 500 to 700 °C. The luminescence results indicated that MgO (100) is a promising substrate for the growth of high-quality of ZnGa@sub 2@O@sub 4-x@Se@sub x@ : Mn@super 2+@ films. The crystallinity and surface roughness of the ZnGa@sub 2@O@sub 4-x@Se@sub x@ : Mn films are highly dependent on the growth conditions, in particular the substrate temperature and the composition ratio of targets. Epitaxial films were obtained on MgO (100) substrates due to the low lattice mismatch between ZnGa@sub 2@O@sub 4@ and MgO. The crystallinity of the films is improved with Se doping. The root mean square surface roughness of these ZnGa@sub 2@O@sub 4-x@Se@sub x@ : Mn@super 2+@ films were found to initially increase from 3.25 nm (x=0.00) to 10.92 nm (x=0.075) then decrease to 8.43 nm (x=0.10) as the amount of Se increases. Incorporation of Se into the ZnGa2O4 lattice led to a remarkable increase of photoluminescence. The highest green emission intensity was observed with ZnGa@sub 2@O@sub 3.925@Se@sub 0.075@ : Mn@super 2+@ films whose brightness was increased by a factor of 3.1 in comparison with that of ZnGa@sub 2@O@sub 4@ : Mn@super 2+@ films. This phosphor is promising for application in flat panel displays.

TF-WeP17 Low-Resistivity Polycrystalline ZnO:Al Thin Films Prepared by Pulsed Laser Deposition, *T. Minami, **H. Tanaka**, K. Ihara, T. Miyata,* Kanazawa Institute of Technology, Japan

Recently, a very low-resistivity Al-doped ZnO (ZnO:Al, AZO) films comparable to ITO films were attained on glass substrates by pulsed laser deposition (PLD) method. In this report, we describe the preparation of low resistivity AZO thin films, including a description of conditions necessary to obtain the low resistivity on the order of 1×10@super -4@@ohm@cm. The AZO films were deposited on glass substrates by PLD using an ArF excimer laser. The deposition was carried out under the following conditions: pressure, 10@super -4@ Pa; substrate temperature, 180-350@super o@C; target-substrate distance, 50 mm; substrate, OA-2 glass; and target, sintered ZnO+Al@sub 2@O@sub 3@(1-3 wt.%). All AZO thin films prepared with a thickness of 200 to 700 nm exhibited an average transmittance above 85% in the visible range. In order to obtain low-resistivity AZO films, it was necessary to optimize the relationship between the target sintering conditions and the deposition conditions. The obtained resistivity decreased as the substrate temperature was increased, reached a minimum at a temperature of about 240@super o@C, and then increased with further increases of the temperature. The decrease of resistivity associated with the increase of temperature to about 240@super o@C resulted from an increase of Hall mobility, whereas the increase of resistivity with higher temperatures resulted from a decrease of carrier concentration. The Hall mobility increase was found to be correlated to an increase of crystallite size, as evaluated from the (0002) x-ray diffraction peak. On the other hand, the Al content in the films increased gradually as the substrate temperature was increased. The decrease of carrier concentration was ascribed to an increase of oxygen content in the films. As another example, the resistivity decreased as the film thickness was increased. As above, the resistivity decrease was also correlated to a Hall mobility increase resulting from an increase of crystallite size. It is concluded that differences in obtainable Hall mobility can be attributed to differences in the crystallinity such as crystallite size.

TF-WeP18 Deposition of ITO Film using the DLC Buffer Layer between Plastic Substrate and ITO Layer, ***M.G. Kim,** H.S. Jeong, Y.W. Seo,* ITM Inc., Korea

Recently organic electro luminescence displays(OELD) using a plastic substrate such as PET and PES are emerging as a strong new candidate for flat panel displays. In this study we make diamond like carbon(DLC) films as a buffer layer between plastic substrate and transparent conducting oxide(TCO). The DLC is characterized by high hardness, flatness, and low gas permeability to moisture and oxygen. The unipolar pulse mode of the power supply(IBP1030, ITM, Inc.) was used on a graphite target, and the

argon-methane mixed gas was used. Indium tin oxide films were deposited on the DLC buffer layer of the plastic substrate in a horizontal single ended in-line sputter system equipped with a pulsed dual magnetron sputter. Two ITO targets of In@sub2@O@sub3@(90 wt%):SnO@sub2@(10 wt%) were used. To understand the properties of the ITO films deposited by pulsed dual magnetron sputtering processes, we investigated the electrical resistivity, film structure, optical transmission, surface roughness, and gas permeability. @FootnoteText@ This work was supported by the Ministry of Science and Technology of the Republic of Korea through the National Research Laboratory program.

TF-WeP19 Deposition of ZnO:Al Gradient Composite Films Using Dual Magnetron Sputtering*, **H.S. Jeong**, H.J. Lee, M.S. Hwang, Y.W. Seo,* ITM Inc., Korea*; S.J. Kwon,* Kyungwon University, Korea

ZnO:Al films with compositional gradient were prepared by dual magnetron sputter deposition technique of in-line sputter system. The films of compositional gradients are achieved by the angle of dual magnetron sources and the moving direction of carrier substrate. The symmetric pulse mode of the power supply(IAP1010, ITM, Inc.) was used to simultaneously sputter a ZnO target and an Al target. We varied the duty of the pulses to control the ratio of ZnO:Al in the thin films. The electrical and optical properties of the films were measured with a surface profiler, an UV/Visible spectrophotometer, a 4-point probe, X-ray diffraction spectroscopy, and atomic force microscopy. Also to observe the depth profile of the compositional gradient film, the film was analyzed by Auger Electron Spectroscopy. The special attention is paid to the surface roughness, electrical resistance, and optical transmittance.

TF-WeP20 ZnO Layers Grown by ns and subps Lasers in Nitrogen Atmosphere*, **M. Jelinek**, L. Soukup,* Institute of Physics AS CR, Czech Republic*; A. Klini,* Foundation for Research and Technology - Hellas (FORTH), Greece*; M. Cernanský, J. Oswald,* Institute of Physics AS CR, Czech Republic*; C. Fotakis, D. Anglos,* Foundation for Research and Technology - Hellas (FORTH), Greece*; R. Zeipl,* Institute of Radio Engineering and Electronics AS CR, Czech Republic*; A. Santoni,* ENEA, Centro Ricerche Frascati, Italy

Thin ZnO films and nitrogen doped ZnO films were grown by subpicosecond (450 fs) and nanosecond (20 ns) KrF pulsed laser deposition (PLD) and by PLD combined with radiofrequency (RF) discharge. Discharge (13.56 MHz) was situated between two RF electrodes placed parallel to plazma plume. Doped ZnO layers were fabricated in mixture of nitrogen and oxygen, for various T@sub s@ and RF power. As substrate fused silica was used. Films were transparent from ~ 350 nm. Highly oriented peaks with FMWH (2? 0.15o was detected by XRD. Films grown in RF discharges were nanocrystalline with crystallites size of 10nm. Hall constant, Hall mobility and resistivity were measured by Van der Pauv method. In dependence on deposition conditions the Hall constant in the range of 1 x 10@super -7@ 2 x 10@super -3@ m@super 3@/As, mobility from 6 x 10@super -2@ to 18.2 cm@super 2@/Vs and resistivity 5.9 x 10@super -5@ to 20 @OMEGA@m were obtained. XPS and WDX results and experimental details will be also presented. @FootnoteText@ The authors thank to the Czech Grant Agency for financial support under contract S1010203.

TF-WeP21 Orientation Selective Epitaxy of CeO@sub 2@(100) or CeO@sub 2@(110) Thin Films on Si(100) Substrates by Magnetron Sputtering with Substrate Bias*, **T. Inoue**, N. Sakamoto, M. Ohashi, A. Horikawa, S. Shida,* Iwaki Meisei University, Japan*; Y. Sampei,* Fukushima Technology Centre, Japan

From studies on the epitaxial growth of CeO@sub 2@ layers on Si(100) substrates using reactive dc magnetron sputtering enhanced with an inductively coupled rf plasma, it is found that the epitaxial CeO@sub 2@ layer with (100) or (110) orientation is selectively grown by controlling substrate bias and plasma power. Although many reports have been made on the growth of CeO@sub 2@(110)/Si(100), recently CeO@sub 2@(100) layers have been reported to grow on atomically cleaned Si(100) surfaces with a 2x1 reconstructed structure in an ultra-high vacuum. Our method has superiority in the requirement of only practical H-terminated surfaces obtained by the usual wet cleaning process. Adopting two step growth method; ultrathin metallic Ce layer deposition at room temperature followed by silicidation process at several hundreds degree C, and subsequent reactive sputtering in an Ar/O@sub 2@ mixture environment using Ce metal target at elevated temperature, the CeO@sub 2@(100) layer epitaxy is attained applying either positive or negative substrate bias of 15 - 20 V, whereas CeO@sub 2@(110) layers are grown without substrate bias. The orientation selection is found to be also dependent on plasma power, in other words the growth rate: the low growth rate leads to (100) orientation and vice versa. This orientation selective epitaxial growth technique will be useful for device applications. Details on the growth parameters of both substrate bias and plasma power and the characterization including RHEED, XRD, XTEM and AFM will be presented.

TF-WeP23 Analysis and Modeling of Low Pressure CVD of Phosphorus-doped Poly-silicon in Commercial Scale Reactor*, **R. Shimizu**, M. Ogino,* Fuji Electric Corporate Research and Development, Ltd., Japan*; M. Sugiyama, Y. Shimogaki,* University of Tokyo, Japan

Poly-silicon is a key material for all kinds of semiconductor devices. The main issue for its deposition technology is how to get the uniform deposition rate and dopant concentration on silicon wafers. In this work, to investigate the poly-silicon CVD mechanisms that control the uniformity in a commercial scale LPCVD reactor, we analyzed the elementary reaction of silane based CVD with the doping gas of phosphine. We used longitudinal type CVD reactor of 6inch manufacturing scale. Under the standard condition, sample wafers were fully charged with a spacing of 4.1mm and the 100% silane and 0.8% phosphine gases with nitrogen carrier gas were introduced to the reactor. The growth temperature was kept 550°C along 700mm length hot-zone and the total pressure was 100Pa in the reactor. On the basis of the diffusion model of chemical species into the wafer-gaps, two precursors were found to contribute the profile of deposition rate, and their sticking probabilities were deduced together with the one of silane. The activation energy of direct surface reaction and gas phase reaction rate constant of silane were experimentally derived from the dependencies of the deposition rate on the growth temperature and wafer spacing, respectively. The diffusion model also gave the relative concentration distributions of the two precursors, which were examined with the elementary reaction analysis based on the Ho's model.@footnote 1@ The effect of phosphine gas on the silane reaction was found to be rather large notwithstanding the small concentration of phosphine gas, as in the studies by others.@footnote 2@ The analysis of this phenomenon will show the detail of the chemical reaction system composed of silane and phosphine gases, and give us a useful predictive model of poly-silicon CVD process. @FootnoteText@ @footnote 1@P. Ho, M. E. Coltrin, and W. G. Breiland, J. Phys. Chem., 98, 10138 (1994)@footnote 2@B. S. Meyerson and W. Olbricht, J. Electrochem. Soc., 131, 2361 (1984).

TF-WeP24 A Study of the Growth Front of Au Policristalline Films and its Relation with the Bulk Structure of the Films*, C. Munuera, J.A. Aznarez, E. Rodriguez,* CSIC, Spain*; A.I. Oliva,* Centro de Investigaciones y Estudios Avanzados del IPN Unidad de Merida, Mexico, Mejico*; M.A. Aguilar, **J.L. Sacedon**,* CSIC, Spain

The surface roughness of Au polycrystalline films with a high (111) texture has been studied using STM images. The growth front has been characterized in samples with thickness from 30 to 1800 nm by measuring the interface width and the coarsening of apparent rounded mounds. In this analysis any isotropic or self-affine hypothesis has been avoided. In addition, the fracture of the thickest films have been analysed by SEM. The films have been obtained by thermal evaporation on native SiO@sub 2@ terminated Si(100) substrates maintained at room temperature. The rate of growth was 1.0 nm/s. The SEM images show a competitive columnar growth for thicknesses from <90nm to 1800 nm. They also show that the mound round shaped structures correspond to the top of the columns. The interface width scaling behaviour obtained from STM images shows two growth regimes, the more advanced starts at 60 nm and corresponds to a constant morphological slope regime.In this advanced stage, the value of the interface width and coarsening scaling exponents are close to 1/3. The exponent values and fracture structures agree with the parametric model of E.V. Albano et al.(PRB 59,7354 (1999)). This model is based on the concept that surface diffusion phenomena controls the bulk structure of the film. Local slopes along the surface profiles are compatible with a significant step down atomic current component, which is necessary to allow the burial of the less competitive columns. The linear boundaries of the surface structure suggest a relaxation of the columnar boundaries to crystalline planes causing the weak hexagonal symmetry observed in the height to height correlation functions.

TF-WeP26 Structure and Properties of Nanolaminate Chromium/ Titanium Oxide Films by Pulsed-DC Reactive Sputtering*, **M.S. Wong**,* National Dong Hwa University, Taiwan, ROC, Taiwan, R.O.C*; M.C. Yang, H.B. Chou,* National Dong Hwa University, Taiwan, ROC*; M.E. Graham,* Northwestern University

The outstanding optical, semiconducting and mechanical properties of titanium and chromium oxides have made them useful for many applications. Ion-assisted, high-rate, reactive, pulsed-dc magnetron

sputtering is used to deposit the nanometer-scale multilayered oxide films. RF substrate bias was applied to induce ion bombardment to modify the structure of the growing films. Various characterization methods such as ellipsometry, Raman spectroscopy, UV-Visible spectroscopy, XRD, SEM, TEM, hardness and stress measurement techniques were used to measure the crystallinity, morphology, optical and mechanical properties of the films. Under suitable conditions, the sputtered metal atoms will react with the partial pressure of supplied oxygen to form stoichiometric and transparent oxide films. Crystalline Cr2O3 and anatase TiO2 were deposited as single-layer films, and as Cr/Ti-oxide multilayers. When the period of the multilayer was below ~7 nm, the multilayer structure was not observed, and Cr-Ti-O alloy films appeared to be formed instead. The energy gaps for Cr2O3, Cr-Ti-O and TiO2 films are ~2.1, ~2.5 and ~3.2 eV, respectively. The Cr2O3 and Cr-Ti-O films possess high hardness, over 20 GPa. The effects of substrate bias and multilayer period on the structure and properties of the oxides will be discussed.

TF-WeP27 Absorption Properties of Thin Film Getter for Impurity Gas in High Vacuum Environment*, **K.C. Kim**, Y.J. Yoon,* Yonsei University, Korea*; S.M. Lee,* Kangwon National University, Korea*; H.K. Baik,* Yonsei University, korea, South Korea

Recently, getters have been widely used in vacuum microelectronics requiring high vacuum. The conventional bulk getters required high temperature activation processes for proper action as a getter, where the device was contaminated by outgassing of different gases, e.g., H@sub 2@O, O@sub 2@, H@sub 2@O, CO, CO@sub 2@. The conventional bulk getter itself was not suitable for the microelectronic devices due to size limitations. As a resolution to such problems, thin film getters, small enough for microelectronic applications, with excellent resonant gas absorption characteristic and requiring no activation, have been fabricated and analyzed. In this research, Zr was used as a getter material, and Ni and Pt were used for catalysis and protection of the getter against oxidation. Thin film getters were fabricated by the introduction of Pt and Ni layers, which exhibited excellent absorption characteristic of impurity gases in high vacuum, without high temperature activation. Realization of thin film getters, by solution of the size limitations of the bulk getters, enabled getter to apply to the microelectronic devices.

TF-WeP28 Control of Crystalline Evolution in Aluminum Nitride Thin Films Deposited by Magnetron Sputtering*, **V.M. Pantojas**, E. García, N.R. Ramos, W. Otaño,* University of Puerto Rico at Cayey

There is considerable interest in the growth of Aluminum Nitride (AlN) thin films for the fabrication of mechanical, optical and surface acoustic wave devices. The properties of polycrystalline AlN thin films depend strongly on the crystallographic orientation and texture of the grains and it is important to have an adequate control of the crystalline characteristics in order to achieve good performance for specific properties. Thin AlN films were prepared using magnetron sputtering. The relationship between the sputtering deposition process parameters and the coating characteristics, and properties, were investigated using Response Surface Design methodology. The deposition pressure, gas composition and substrate temperature were used as factor levels to characterize the response of the most important characteristics, crystallinity and stoichiometry. The evolution of crystal orientation in AlN thin films was analyzed using an x-ray diffraction system with a general area detector. The two dimensional, position sensitive area detector allows for fast collection of the x-ray scattering data and provides the unique capability of directly imaging the orientation of the diffraction pattern. The degree of crystal orientation of the AlN samples varied from completely polycrystalline to highly oriented depending on the deposition conditions. In order to quantify the degree of crystallinity, an integration method was used. The three main peaks of hexagonal AlN were integrated along a rectangular slit at the center of the detector to quantify the degree of in-plane orientation and a texture coefficient was calculated for each crystal orientation.

TF-WeP29 Synthesis and Electrochemical Characteristics of Boron Carbon Nitride Films Deposited by Magnetron Sputtering*, **E. Byon**,* Korea Institute of Machinery and Materials, Korea*; M. Son,* Samgsung Techwin Co. Ltd, Korea*; N. Hara, K. Sugomoto,* Tohoku University, Japan*; S.-K. Kwon,* Korea Institute of Machinery and Materials

Boron-Carbon-Nitrogen (BCN) system is an attractive ternary material since it has not only an extremely high hardness but also a number of other prominent characteristics such as chemical inertness, and low thermal expansion. In this paper, we prepared ternary BC@sub x@N@sub y@ films on Si by r.f. magnetron sputtering of boron and graphite targets. With increasing of the graphite target power, FTIR and XPS showed that the structure of the BC@sub x@N@sub y@ films was changed from the cubic BCN to the hexagonal BCN. The corrosion resistance of BCN thin films in aqueous solution was investigated. BCN films with different composition were deposited on a platinum plate in the thickness range of 150-280 nm. In order to understand effect of pH of solutions on the corrosion resistance of the film, BC@sub 2.4@N samples were immersed in 1 M HCl, NaCl and NaOH at 298 K respectively. BCN films with different carbon content were also immersed at 1 M NaOH solutions to investigate effect of chemical composition on the corrosion resistance. The thinning rate of the film thickness was measured at various potentials in 1 M NaCl by using in-situ ellipsometry. From the results, the corrosion resistance of B@sub 1.0@C@sub 2.4@N@sub1.0@ films were NaOH>NaCl>HCl in that order. With increasing carbon content in BCN films, the corrosion resistance of BCN films is enhanced. The lowest corrosion rate was obtained for B@sub 1.0@C@sub(3.2-4.4)@N@sub 1.2@ films. The anodic polarization in 1 M H@sub 2@SO@sub 4@, 1 M HCl and 1 M NaCl showed similar behavior with Pt substrate. Hydrogen overvoltage in the cathodic region was higher than Pt substrate. No active dissolution of the films was occurred in the range of â€“0.2 V to 0.1 V of potential in 1 M NaOH, but the film thickness reduced with increasing potential. The current density of the films is directly proportional to the dissolution rate of B1.0C2.4N1.0 film over 0.2 V of potential.

TF-WeP30 Mechanical Properties of Ultranano-, Nano- and Poly-crystalline Diamond Films and Membranes*, **A. Moon**, T.A. Grotjohn,* Michigan State University and Fraunhofer Center for Coatings and Laser Applications*; M.K. Yaran, T. Schuelke,* Fraunhofer Center for Coatings and Laser Applications*; D.K. Reinhard, J. Asmussen,* Michigan State University and Fraunhofer Center for Coatings and Laser Applications

The mechanical properties of polycrystalline materials can vary substantially depending on the material's grain size, surface morphology and grain boundary properties. In the case of diamond films the crystal grain size and surface morphology can range from ultrananocrystalline (grain sizes of less than 10nm) to nanocrystalline (grain sizes of 10's nm) to polycrystalline (grain sizes of 100's nm to microns). The Young's modulus of films and the deflection characteristics and fracture strength of membranes are characterized in this study for a range of diamond films/membranes deposited with grain sizes ranging from ultrananocrystalline to polycrystalline. The Young's modulus of diamond films with thickness from 0.1 µmm to 2 µmm is measured on the substrate by using a pulsed laser to generate surface acoustic wave which are evaluated by Fourier analysis. The Young's modulus and membrane deflection characteristics are characterized as function of the thickness of the film, film nucleation/seeding technique, grain size, surface morphology, and synthesis chemistry. Measured Young's modulus values were as high as 900-1000 GPa.

TF-WeP31 Improvement of Corrosion Resistance of Transparent Conductive Multi-layer Coatings Consisting of Silver Layers and Transparent Metal Oxide Layers*, **K. Koike**, F. Yamazaki, T. Okamura, S. Fukuda,* Mitsui Chemicals, Inc., Japan

We have investigated transparent conductive multi-layer sputter coatings consisting of silver (Ag) layers and transparent metal oxide layers. We could expect versatile applications for the multi-layer sputter coatings, such as a transparent electrode and a transparent electromagnetic waves shield. The multi-layer sputter coatings including the silver layer is very sensitive to surrounding atmosphere. For example we would find discolored points on the multi-layer sputter coatings, possibly caused by a silver atom migration in silver layers, after an exposure test. In our investigation, we modified a top surface of the multi-layer sputter coatings with transition metals to improve a corrosion resistance of the multi-layer coatings. Specifically we deposited the transition metals with 0.5 to 2nm thickness on the top surface of the multi-layer coatings by sputtering. We chose indium tin oxide (ITO) as the transparent metal oxide. We applied the multi-layer sputter coatings of 7 layers formed on a polyethylene terephthalate (PET) film substrate. A cross-sectional structure of the film with the multi-layer coatings is PET film / ITO / Ag /ITO / Ag / ITO / Ag / ITO. We evaluated the corrosion resistance by a salt water immerse test. In the test we immerse the film with multi-layer coatings into salt water, and then evaluate an appearance, a transmittance and an electrical resistance of the multi-layer coatings. We investigated several transition metals as the modifying material, and we found that titanium and tantalum drastically improved a resistance of the multi-layer coatings against the salt water without a significant decline of a transmittance. We also investigated a relation between an elapsed time after the depositing the modifying materials and the resistance against the salt water. Further we investigated effects of a

heat treatment and an oxide plasma treatment on the resistance against the salt water.

TF-WeP32 Anisotropic Film Growth during PVD on Substrates in Planetary Motion

G.C.A.M. Janssen, Delft University, The Netherlands; ***J.-D. Kamminga,*** NIMR, The Netherlands; *P.F.A Alkemade,* Delft University, The Netherlands; *S.Yu. Grachev,* NIMR, The Netherlands

Chromium films were deposited in an industrial PVD machine, designed to coat 3-D objects. The machine is equipped with a substrate table that allows a planetary motion of the substrates in front of the target. Films with thickness ranging from 30 nm to 10 Âµm were deposited on Si wafers. All films are under tensile stress. The thinner films exhibit a 110 fiber texture. The curvature of the substrates with the thinner films is radially symmetric. Therefore the stress is radially symmetric. The thicker films exhibit an in plane "single crystal" type texture. For these films the stress is no longer radially symmetric. The curvature of the wafers with the thicker films is distinctly different in the plane of planetary motion and perpendicular to that plane. A SEM top view of the thicker films shows an anisotropy in grain shape. We discuss the coinciding occurrence of anisotropy in stress and microstructure.

TF-WeP33 Combined RF Magnetron Sputtering and Ion Implantation for the Synthesis of Silicon Carbonitride Thin Films

M. Bruns, *H. Lutz,* Forschungszentrum Karlsruhe GmbH, Germany; *M. Rudolphi, H. Baumann,* Universitaet Frankfurt, Germany

Silicon Carbonitride thin films have been the subject of great interest in recent years due to the expected improvement of surface properties for a lot of applications. Various precursor based techniques have been employed to synthezise the pure materials. However, most of these efforts result in amorphous films or tiny crystals embedded in amorphous matrices of deficient nitrogen content and considerable hydrogen and oxygen impurities, respectively. In contrast, RF magnetron sputtering as well as ion implantation have been proven suitable means to achieve high-purity ternary systems of up to 57 at.% nitrogen content, e.g. Si@sub 2@CN@sub 4@. However, carbon rich compounds (i.e. SiC@sub 2@N@sub 4@) are not attainable on this way. For that the combination of both means seams to be a promising approach. Defined and reproducible Si/C ratios within the films can be obtained using co-sputter targets of different Si/C areas. In a second step surface modification by high fluence implantation of @super 15@N ions into these Si-C films result in suitable nitrogen content up to the theoretical amount. Severalfold implantation at different energies calculated from Monte-Carlo-simulations enable us to synthesize layers with homogeneous element depth-distribution up to the surface. @paragraph@The chemical composition of the Si-C-N films was characterized by means of X-ray photoelectron spectroscopy (XPS). In addition, Auger electron spectroscopy (AES), Fourier transform infrared spectroscopy (FTIR), and Raman spectroscopy were used to achieve a comprehensive characterization. For quantification XPS and AES data were calibrated with absolute concentration values from non-Rutherford backscattering spectrometry (n-RBS). Resonant nuclear reaction analysis (NRRA) provides non-destructive depth profiles of @super 15@N. The morphology after subsequent annealing was studied by means of X-ray diffraction (XRD) and transmission electron microscopy (TEM).

Magnetic Interfaces and Nanostructures
Room 316 - Session MI+TF-WeA

Magnetic Thin Films
Moderator: D.P. Pappas, National Institute of Standards and Technology

2:00pm **MI+TF-WeA1 Electrodeposition of Epitaxially Grown Fe Films on n-type GaAs***, C. Scheck, **Y.-K. Liu,*** The University of Alabama*; G. Zangari,* University of Virginia*; R. Schad,* The University of Alabama

Epitaxial growth of Fe thin films on both n-type GaAs(001) and n-type GaAs(011) substrates have been demonstrated using Molecular Beam Epitaxy (MBE) in the past. Indeed, Fe and GaAs lattice constants match very well leading to easy epitaxial growth. However, special surface preparation or growth at elevated temperature were needed to obtain good quality films. Also diffusion and segregation of substrate material (As) at the surface, characteristic of intermixing at the interface, were observed on MBE grown films. Electrodeposition (ECD) technique on the contrary is an equilibrium process which thus releases much less energy per absorbed atom than other deposition techniques (MBE). This allows preparation of chemically sharp interfaces (i.e. no intermixing) which otherwise show a high degree of reactivity and interdiffusion. We reported for the first time the epitaxial growth of high quality Fe thin films on both n-type GaAs(001) and n-type GaAs(011) substrates using ECD. Two different electrolytes FeSO4 and FeC§¤2 solutions 0.1M were used at pH 2.5. Results from X-Ray Diffraction (XRD) show Fe(001)[110]//GaAs(001)[110] and Fe(011)[100]//GaAs(011)[100] as the primary epitaxial relations similarly to Fe films grown by MBE. These films' in-plane magnetic anisotropy is related to the crystalline structure. Their coercivity Hc is around 30-100 Oe.

2:20pm **MI+TF-WeA2 Epitaxial Growth of Ferromagnetic Fe Overlayers on CH@sub 3@CSNH@sub 2@ - Passivated GaAs(100)-S(2x1) Reconstruction***, **E.D. Lu**, H.T. Johnson-Steigelman, P.F. Lyman,* University of Wisconsin, Milwaukee

Epitaxial growth of ferromagnetic metallic Fe overlayers on thioacetamide (CH@sub 3@CSNH@sub 2@)-passivated GaAs(100)-S(2x1) reconstructed surfaces at room temperature (RT) has been investigated by low energy electron diffraction (LEED), x-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES). Prior to Fe deposition, GaAs(100) wafers were passivated by CH@sub 3@CSNH@sub 2@ solution then annealed between 320°C and 450°C under UHV. A clear (2x1) reconstructed LEED pattern with around 1 monolayer (ML) sulfur coverage resulted. Upon deposition of Fe at RT, epitaxial bcc(100) Fe overlayers could be grown from 3 to 40 ML. XPS and AES have revealed that only an initial interface reaction (<4ML) takes place between the evaporated Fe overlayer and GaS sulfide passivation layers. Upon annealing, the Fe/S-GaAs(100) heterostructure appears stable up to 320°C; solid state reaction and/or interdiffusion of the layers starts at higher annealing temperatures, becoming severe by 450°C. Nonetheless, a LEED pattern is observed even after intermixing. These results indicate that the S passivation layer may inhibit Ga and As outdiffusion at modest substrate temperatures, and may thus suppress or reduce the formation of an anti-ferromagnetic Fe@sub 2@As dead layer or other unfavorable Fe@sub 3@Ga@Sub 2-x@As@sub x@ phases.

2:40pm **MI+TF-WeA3 On the Magnetic Properties of Ultra Thin Epitaxial Fe Films on GaAs(001)***, S.A. Morton,* Lawrence Livermore National Laboratory*; J.R. Neal, M. Spangenberg, T.-H. Shen,* University of Salford, UK*; A.E.R. Malins, E.A. Seddon,* CLRC Daresbury Laboratory, UK*; D. Greig,* University of Leeds, UK*; J.A.D. Matthew,* University of York, UK*; **G.D. Waddill**,* University of Missouri, Rolla*; J.G. Tobin,* Lawrence Livermore National Laboratory

The magnetic properties of epitaxial Fe films on GaAs have attracted considerable interest in recent years because of their potential for use as spin injection sources for spintronic devices; however, previous studies have been unable to demonstrate a magnetic signature in films with thicknesses below approximately 5 ML. A number of possible explanations for this observation have been proposed such as the presence of a magnetically dead interfacial layer of Fe2As. However, by measuring the thickness and temperature dependence of the Fe 3p core level magnetic linear dichroism signal we have shown that such films are indeed ferromagnetic but that their Curie temperature is substantially below room temperature. For instance a Tc of approximately 240K was measured for thin films with a nominal thickness of 0.9 nm. Furthermore the values of the Curie temperature in this thickness regime have been shown to be extremely sensitive both to initial substrate conditions and to the overall film thickness; an increase in the thickness of 0.35nm results in a Curie temperature above room temperature. These measurements have been complemented with spin resolved and angle resolved measurements of the Fe/GaAs valence band and core levels; together with ex-situ XRD and SPM studies of sample growth. The origins of the evolving magnetic behavior of Fe films on GaAs(001) from 1-50ML is discussed in terms of the spin dependent electronic band structure and the stoichiometry and growth morphology.

3:00pm **MI+TF-WeA4 Correlated Structural and Magnetization Reversal Studies on Epitaxial Ni Films Grown with MBE and with Sputtering***, **Z. Zhang**[1], R.A. Lukaszew,* University of Toledo*; A. Zambano,* Michigan State University*; C. Cionca,* University of Michigan, Ann Arbor*; D. Walko,* Argonne National Laboratory*; E. Dufresne,* University of Michigan, Ann Arbor*; M. Yeadon,* National University of Singapore, Singapore*; R. Clarke,* University of Michigan, Ann Arbor

The study of epitaxial magnetic thin films is important to understand structure-property correlations. We have studied the correlation between film structure and azimuthal dependence of the magnetization reversal on (001) epitaxial Ni films grown on MgO substrates with two different deposition techniques: molecular beam epitaxy (MBE) and DC magnetron sputtering . The films were grown and annealed in-situ under identical conditions. The magnetization reversal was investigated using MOKE. The coercive field in the sputtered films exhibits 4-fold symmetry as expected for a crystal of good epitaxial quality. The MBE grown films exhibit an additional uniaxial symmetry superimposed to the four-fold symmetry. We note that films of the same thickness made with these two deposition techniques, exhibited the same average coercivity. We performed high-resolution XRD at the Advanced Photon Source (ANL) in order to establish similarities and differences in the structure of the films. Both types of films exhibit epitaxial growth and very good crystalline quality with no indication of strain, and don't exhibit fundamental structural differences. The main difference between the films was the surface morphology. STM images of the surface of the MBE grown films indicated self-assembled periodic stripe nano-patterning, while STM images of the sputtered films didn't exhibit any regular patterning of the surface. Cross sectional TEM studies performed on the films will be correlated with the surface morphology and with the magnetic anisotropy.

3:20pm **MI+TF-WeA5 Adsorption-induced Giant Stress and Surface Relaxation in Ni/W(110)***, **H.L. Meyerheim**, D. Sander, R. Popescu,* Max-Planck-Institut f. Mikrostrukturphysik, Germany*; O. Robach, S. Ferrer,* ESRF, France*; J. Kirschner,* Max-Planck-Institut f. Mikrostrukturphysik, Germany

Surface stress has been recognized as a decisive factor, which determines a variety of phenomena like self-assembled pattern formation on the nano-scale, shape evolution of nano-objects and surface reconstruction. Our work reveals an important aspect of adsorbate-induced stress which might be of key importance for the general understanding of stress-strain relations in the monolayer range. We identify an intimate correlation between structure and stress from an in-situ combination of surface x-ray diffraction and stress measurements. We show that one atomic layer of Ni induces substantial lateral shifts of the top W-atoms of up to 0.5 Å. At this coverage we measure an anisotropic change of the surface stress induced by Ni. The experiments were carried out at the ESRF in Grenoble (France). Surface stress was measured by the crystal curvature technique.@footnote 1@ From earlier experiments@footnote 2@ it is known that Ni forms a c(1x7) superstructure on W(110) at a coverage above 0.8 monolayers (ML, 1ML=1.41x10@super15@ atoms/cm@super2@). Up to 0.8 ML the stress measurements indicate compressive stress along [001] and tensile stress along [1-10]. The emergence of the c(1x7)-structure coincides with the formation of compressive stress along [1-10]. The x-ray analysis shows that the Ni-atoms form a distorted densely packed hexagonal adlayer. However, the most important and astonishing aspect is the pronounced shifts (up to 0.5 Å) of the first layer W-atoms out of their bulk positions along [1-10]. The structure consists of alternating Ni-W-Ni-chains running along [1-11]. In conclusion we have measured a substantial adsorbate-induced relaxation in a system, which is generally considered to be structurally inert upon adsorbate deposition. There is a striking correlation between the onset of lateral shifts of atomic positions and compressive surface stress. @FootnoteText@@footnote 1@D. Sander et al. Rev. Sci. Instr. 66, 4734 (1995); @footnote 2@D. Sander et al. Phys. Rev. B57, 1406 (1998).

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[1] **Falicov Student Award Finalist**

3:40pm **MI+TF-WeA6 Ion Deposited Co and DLC Films Generated from Laser Excitation**, ***F.J. Cadieu***, *L. Chen,* Queens College of CUNY

A modified pulsed laser deposition system has been used to deposit films from ions generated due to the impact of 248 nm excimer laser pulses. A synchronously pulsed magnetic field coil with a wide entrance throat and a tapered bore in conjunction with a set of permanent magnets has been used to capture and concentrate the ion and electron beam flux under vacuum conditions onto the substrates. Co and other films such as diamond like carbon, DLC, are strongly adherent when deposited onto even room temperature substrates. The ion collection and concentration factor is such that Co films deposited on glass to an optical density of 3.0, approximately 7 nm thickness, with coil pulsing and magnets only exhibited an optical density of 0.10 without coil pulsing and magnets for the same number of laser pulses. Films have been characterized by x-ray reflectivity, x-ray diffraction, scanning electron microscopy, SEM, and magnetoresistivity measurements when combined with other magnetic layers. Over an order of magnitude intensity modulation has been observed in x-ray reflectivity measurements for Co films, 7-15 nm thickness, deposited onto R-plane sapphire substrates and onto Si (111) substrates. In contrast to this Co films simultaneously deposited onto A-plane sapphire substrates hardly exhibited x-ray reflectivity fringes indicating a large surface roughness. Similarly DLC films deposited onto (111) Si substrates exhibited order of magnitude intensity modulations indicating a very small surface roughness. X-ray reflectivity measurements of DLC, Co layers on (111) Si are used to show the conditions necessary to make smooth and hence insulating DLC films onto the metallic layer. SEM measurements indicated smooth films were obtained with no discernible particulates. X-ray pole figures are used to characterize single crystal films versus polycrystalline growth modes.

4:00pm **MI+TF-WeA7 Ultrathin Magnetic Layers Electrodeposited on Au(111)and Related Technological Substrates**, ***P. Allongue***, *F. Maroun,* CNRS, France*; J.E. Schmidt, A. Gundel,* UFRGS*; M.L. Munford,* UFSC*; T. Devolder,* IEF, France **INVITED**

Magnetic properties such as interface perpendicular anisotropy (PMA) is strongly depending on the coupling between the substrate, generally a noble metal, and the ultrathin ferromagnetic layer. Both the film and interface structures are also critically influencing the strength of PMA. We recently showed that electrodeposited Cu/Co/Au(111) structures exhibit strong PMA due to an excellent 2D epitaxial growth of cobalt and some residual elastic stress at the interface. This talk will be divided into two parts. In the first one, I will present in situ structural and magnetic characterizations of M / Au(111) ultrathin layers, with M = Co, Ni and Fe using STM, EXAFS and real time in situ magnetic characterizations (AGFM and PMOKE) with sub-monolayer sensitivity. It will be shown that the latter measurements, performed in the electrolytic environment, yield new information regarding the relationship between the deposition conditions (potential, pH etc.) and the magnetic state of layers. While Co and Fe layers are ferromagnetic at submonolayer coverages, results show that Ni layers become ferromagnetic only after the deposition of 5-6 monolayers. By proper adjustment of the deposition condition, so as to avoid dramatic H-incorporation, ultrathin ferromagnetic Ni layers may however be prepared. The strength of PMA at Co/Au(111) layers will be discussed in details. The second part of the talk will address the deposition of cobalt on epitaxial Au(111) layers on vicinal H-Si(111) surfaces. We are able to prepare ultraflat Au films or Au films consisting in nm-sized dots aligned along the step edges. As a consequence, we may prepare Co/Au/Si films with quite different magnetic properties. For instance, in the case of Au dots in plane anisotropy is obtained and it is possible to vary the coupling between lines of Co dots by changing the Si miscut. These results will be discussed in details at the conference.

4:40pm **MI+TF-WeA9 The Effect of Interlayer Coupling to the Magnetic Phase Transition of Thin Films**, ***C. Won***, *Y.Z. Wu,* University of California at Berkeley*; A. Scholl, A. Doran,* Lawrence Berkeley National Laboratory*; N. Kurahashi, H.W. Zhao, Z.Q. Qiu,* University of California at Berkeley

Magnetic phase transition of two-dimensional systems is one of the intensively studied topic in condensed matter physics. One of the basic questions on this subject is how the addition of the interlayer coupling changes the magnetic phase transition. The coupled magnetic sandwiches of Co/Fe/Ni/Cu(100) and Co/Cu/Ni/Cu(100) were investigated by photoemission electron microscopy(PEEM). Element-specific magnetic domain images were taken at room temperature to reveal the critical thickness at which the magnetic phase transition occurs. The phase diagrams with thickness of each magnetic film were constructed under a few selected coupling conditions. The results shows three different types of magnetic phase transitions depending on the relative ferromagnetic film thickness. If the magnetic orders of two magnetic films are similar, both films undergo magnetic phase transition simultaneously. This means the lost of magnetic order of one film can be compensated by the interlayer coupling with the other magnetic film and two magnetic films are highly correlated each other in the interlayer coupling strength. Other two phase-transitions happened when one film is coupled with the other film that is too thin to have magnetism even with the help of coupling or that is ferromagnetic already by its own magnetic order. The difference of the critical thickness for these two cases shows that interlayer coupling increases Curie temperature. The strength and sign of coupling was changed to see how the coupling strength changes this behavior and we found that not the sign but the coupling strength has main role in the phase transition. And Monte-Carlo simulations based on 2 dimensional Ising model were performed to explain this experimental results.

5:00pm **MI+TF-WeA10 Biased Target Ion Beam Deposition of GMR Multilayers**, *H.N.G. Wadley,* ***X.W. Zhou***, *J.J. Quan, S. Subha,* University of Virginia*; T. Hylton, D. Baldwin,* 4Wave, Inc.

Detailed atomistic simulations have identified the preferred deposition conditions for growing the ideal atomic structures that maximize the performance of giant magneto resistive (GMR) multilayers. They reveal that increasing the velocity (energy) of condensing atoms or assisting ion fluxes flattens interfaces, but promotes atomic interlayer mixing. The maximum magneto resistance is believed to occur for the lowest combination of interfacial roughness and interlayer mixing. Low values of this metric have been predicted to occur using a constant intermediate energy of a few electron volts throughout the growth process. However, the lowest values of the metric have been predicted to occur when a modulation of the energy during deposition of each material layer is used. It is difficult to implement such processes in a conventional PVD or ion beam system. We have developed a biased target ion beam deposition system to overcome these difficulties and report its design and the characteristics of the spintronic devices grown with it.

Thin Films
Room 326 - Session TF+MM-WeA
Sensors, Smart Films and Functional Materials
Moderator: C.H. Stoessel, Consultant

2:00pm **TF+MM-WeA1 CMOS-Based Microsensors**, ***O. Brand***, Georgia Institute of Technology **INVITED**

CMOS-based microsensors combine, on a single chip, the necessary transducer elements and integrated circuits. This way, the microsensors benefit from well-established fabrication technologies and the possibility of on-chip circuitry. Besides sensor biasing and signal conditioning, added on-chip functionality, such as calibration, self-testing, and digital interfaces, can be implemented. A number of microsensors, including magnetic field and temperature sensors, are completely fabricated within the regular CMOS process sequence. A far larger number of microsystems can be realized by combining CMOS or BiCMOS technology with compatible micromachining and thin film deposition steps. These additional fabrication steps are performed either before, in-between, or after the regular CMOS process sequence. Commercially available examples include pressure sensors, accelerometers, gyroscopes, humidity sensors, mass flow sensors, and imaging devices. In the first part, the paper summarizes major technological approaches to CMOS-based sensors. In the second part, a packaged CMOS-based chemical microsystem, developed at ETH Zurich, Switzerland for the detection of volatile organic compounds in air is highlighted. On a single chip, the microsystem combines a sensor array featuring three different sensing principles with circuitry for sensor biasing, signal read-out, analog-to-digital conversion, and digital interfacing. The chemical microsystem is fabricated using an industrial CMOS technology in combination with post-processing bulk-micromachining to release the micromechanical sensor structures. After packaging the microsystem using flip-chip technology, the three sensor structures are coated with chemically sensitive polymer films. Absorption of volatile organic compounds in the polymer films results in a change of the (physical) film properties, such as the mass, dielectric constant, or temperature, which is then recorded by the underlying sensor structure.

2:40pm **TF+MM-WeA3 Behavior of Thin Ionic Liquid Films Studied with Atomic Force Microscopy**, ***J.J. Nainaparampil***, *B.S. Phillips,* AFRL/MLBT*; K.C. Eapen,* University of Dayton Research Institute*; J.S. Zabinski,* AFRL/MLBT

Ionic liquids (IL's) represent a new class of solvents having the character of molten salts. They have no detectable vapor pressure, are moisture, air and temperature stable and therefore are excellent solvents. Most of these IL's consists of cations such as different alkyl imidazolium or alkyl pyridinium ions and anions such as BF@sub 4@, PF@sub 6@, N (CF@sub 3@SO@sub 2@)@sub 2@, CF@sub 3@SO@sub 3@. In this work, crystals of alkyl imidazolium+ PF@sub 6@ are dissolved in water or acetonitrile to form 0.2% to 0.5% solutions and deposited on Si surface to form thin films. Atomic force microscope working in non-contact mode capable of providing biased tip lithography is used to characterize these films. It is observed that scanned films give rise to certain geometrical structures that are repeated in recrystallized surfaces. A biased AFM tip is used to mobilize these structures to form other complex structures. It is noted that the same solution, when used as a lubricant in sliding contacts gives rise to drastic friction reduction compared to other lubricants. These IL's were used to lubricate a micro electro mechanical system (MEMS) electro static output motor. Results from MEMS endurance tests and an interface model that is based on the AFM study will be presented. Electro-migration of crystallites and adhesion of transfer films affect the friction and durability significantly.

3:00pm **TF+MM-WeA4 Bilayer Transition-edge Sensors for X-ray Calorimeter and Infrared Bolometer Arrays**, ***J.N. Ullom***, *J.A. Beall,* National Institute of Standards and Technology*; J. Beyer,* PTB, Guest Researcher NIST*; S. Deiker, W.B. Doriese, G.C. Hilton, K.D. Irwin, C.D. Reintsema, L.R. Vale,* National Institute of Standards and Technology **INVITED**

Microcalorimeters and bolometers made from thin superconducting films cooled to temperatures near 100 mK have made dramatic progress in recent years. These devices provide an order of magnitude improvement in energy resolution over existing semiconductor x-ray sensors and are likely to be used in upcoming astronomical instruments spanning the spectrum from x-ray to millimeter wavelengths. The sensitivity of these devices is derived from the low heat capacities and thermal conductivities possible near 100 mK and from the strong dependence of resistance on temperature in the superconducting-to-normal transition. Our devices are made from bilayers of a normal metal and a superconductor. Use of a bilayer allows the transition temperature and resistivity of the sensors to be precisely controlled. In this talk, we describe recent progress towards kilopixel sensor arrays using multiplexed SQUID readout. We are building arrays of x-ray microcalorimeters for two applications: energy-dispersive x-ray spectroscopy on scanning electron microscopes and for the upcoming NASA satellite Constellation-X. We are building arrays of submillimeter bolometers for the SCUBA-2 camera on the James-Clerk Maxwell Telescope on Mauna Kea. At this time, the measured noise in both microcalorimeters and bolometers approaches but does not equal the value predicted from simple thermodynamics. We will present measurements of this excess noise and describe recent mitigation efforts.

Thin Films
Room 329- - Session TF1-WeA
Thin Film - Based Combinatorial Methods

Moderator: J. Kidder, Vermont Technical College

3:40pm **TF1-WeA6 Combinatorial Approach to Functional Thin Film Materials**, ***I. Takeuchi***, University of Maryland **INVITED**

In this talk, I will describe the combinatorial materials research methodology we have developed using thin film techniques targeting a variety of functional materials. The basic idea of combinatorial thin film deposition systems is to create controlled compositional variation across given chips. Versatile compact combinatorial pulsed laser deposition systems are used for pursuing metal oxide systems. For studying metallic alloys, an UHV co-sputtering system is used to fabricate natural composition spreads. In this system, three magnetron guns are placed in a parallel geometry in order to maximize the coverage of ternary compositional phase space on three inch wafers. The spreads are primarily used for rapidly exploring novel phase space of magnetic materials. Various high-throughput characterization tools are used for rapid characterization of thin film combinatorial libraries and composition spreads. They include quantitative scanning microwave microscopes for studying dielectric/ferroelectric and resistive materials, scanning SQUID microscopes for mapping of magnetic properties and a scanning X-ray diffractometer for structural characterization. We also implement micromachined device libraries such as arrays of cantilevers for investigation of smart materials. Our studies have led to identification of novel compositional regions of ferromagnetic shape memory alloys.

4:20pm **TF1-WeA8 Preparation of Ternary Alloy Libraries for High-throughput Screening of Material Properties by Means of Thick Film Deposition and Interdiffusion: Benefits and Limitations**, ***A. Rar***, Oak Ridge National Laboratory and The University of Tennessee*; E.D. Specht,* Oak Ridge National Laboratory*; E.P. George, G.M. Pharr,* Oak Ridge National Laboratory and The University of Tennessee

Numerous techniques have been developed for making ternary alloy libraries for combinatorial materials development. Two popular approaches for synthesizing films are: (i) co-deposition from 3 different pure element sources and (ii) preparation of multilayer thin films with thickness gradients in different directions for each component, followed by annealing of the films to achieve local alloying. However, both methods have limitations. In the first, the elemental distribution is non-linear, the film thickness is not constant, and regions with low concentrations of one or more elements are difficult to achieve. In the second approach, synthesis of thick films may be a problem because of the large numbers of layers required. One possible solution is to deposit a single relatively thick layer for each element, followed by annealing to achieve alloying. This approach was examined for the Ni-Fe-Cr ternary system. Spatially resolved alloy properties were compared with well known structural properties by means of rapid XRD mapping with synchrotron radiation. Specimens were prepared by depositing films onto sapphire substrates with an e-beam evaporation system. After deposition, the layers were interdiffused by annealing in different environments. The quality of the resulting specimens was examined using cross sectional SEM, electron microprobe analysis, angular resolved x-ray fluorescence, and XRD. The main problems were encountered during annealing. Selection of annealing temperatures and times that could be used to produce good interlayer diffusion without Cr evaporation or Kirkendall voiding proved difficult. In addition, there was a tendency to form chromium oxide at the surface. Despite these problems, an isothermal section of the ternary phase diagram was reasonably well reproduced.

4:40pm **TF1-WeA9 Combinatorial Pulsed Laser Deposition for Investigation of Metal Oxide Systems**, ***K.S. Chang***, University of Maryland, College Park

Pulsed laser deposition (PLD) is an efficient technique for fabrication of a variety of thin film materials especially metal oxides. We have developed a compact combinatorial PLD system. The heart of the system is contained in a portable combinatorial thin film deposition flange which can be fitted into any physical vapor deposition system. The eight-inch flange features an automated two-dimensional shutter/mask system and a rotatable substrate heater which can go up to 800 ÂºC. Spatially selective shadow depositions are carried out by controlling the motion of the shutter/masks which glide over a mounted substrate during and in between depositions. Different designs of discrete combinatorial libraries and continuous composition spreads can be achieved by cutting different apertures on the replaceable masks made of stainless steel sheets. SnO2 based semiconductor gas sensor libraries consisting of 16 discrete compositions have been fabricated, and their operation as electronic noses were successfully tested. By monitoring the number of laser pulses, one can control the deposition of materials at atomic layer level. This layer-by-layer technique can be used for epitaxial growth of continuous composition spreads with controlled compositional variation across chips. Some of the materials we have looked at include ferroelectric BaTiO3-SrTiO3 composition spreads where continuous change in microwave properties were studied and MgO-ZnO composition spreads which can be used for construction of solar blind multi-channel UV detector arrays.

5:00pm **TF1-WeA10 Gadolinium-doped Yttrium Aluminum Garnet Ultraviolet Emitting Materials Deposited by rf Reactive Magnetron Sputtering**, ***Y. Deng***, *J.D. Fowlkes,* University of Tennessee*; J.M. Fitz-Gerald,* The University of Virginia*; P.D. Rack,* University of Tennessee

Gadolinium is known to radiate in the ultra-violet region at ~ 312nm and 275nm due to intra-band 4f transitions when suitably doped in oxide host materials. To investigate the ultraviolet Gd emission, thin films of gadolinium-doped with yttrium aluminum garnet (YAG:Gd) have been deposited by rf reactive magnetron sputtering. The parameters in this work include RF power, substrate temperature, O2 partial pressure ratio and annealing temperature. An optimized combinatorial process has been

obtained by statistical analysis on a factorial design of experiment. The structure and composition of the films have been characterized by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). The crystallinity of the films has been investigated by X-ray diffraction (XRD). The effect of Gd doping level on the cathodoluminescent (CL) properties of the films has also been studied and has been correlated to the chemical and microstructural properties of the films.

Thin Films
Room 329 - Session TF2-WeA
Optical Thin Films and Photovoltaics II

Moderator: R. Sargent, OCLI

2:00pm **TF2-WeA1 Magnetron Sputtering for II-VI Semiconductor PV Materials***, **A.D. Compaan,*** University of Toledo **INVITED**

The deposition of polycrystalline, thin-film, II-VI semiconductors such as CdTe is possible by many different techniques, but the use of a plasma-based method such as magnetron sputtering can have significant advantages. In this paper we review recent advances in the fabrication of CdS/CdTe cells using rf planar magnetron sputtering and discuss some of the advantages that accrue from the use of sputtering in this class of materials. Some of these advantages take on increased relevance as the polycrystalline thin-film community begins to address issues related to the challenges of fabricating high efficiency tandem cells with efficiencies over 25%. Recently we have achieved improved sputtered cell performance (Voc= 814 mV, Jsc=23.5 mA/cm2, fill factor = 73.25, and efficiency = 14.0%) from superstrate cells based on window layers with sputtered ZnO:Al. In addition we have used reactive sputtering for the deposition of nitrogen-doped layers of p-ZnTe for possible use in transparent back contacts to CdTe. We shall also discuss recent results in the sputtering of wider and narrower bandgap alloys of CdTe with Zn, Mn, and Hg, and in the fabrication of cells with very thin CdTe layers having relatively little loss in performance and stability. Finally, the use of magnetron sputtering permits the fabrication of flexible thin-film cells on temperature-sensitive substrates such as polyimides. Work supported by the National Renewable Energy Lab and the U.S. Air Force.

2:40pm **TF2-WeA3 The Effects of Cu and Cl at the Device Junction on the Performance of CdTe-CdS Photovoltaic Cells***, **T.J. Bukowski**, D. Albin, J. Pankow, S.E. Asher,* National Renewable Energy Laboratory

Current CdTe photovoltaic device design utilizes processing steps that incorporate both Cu and Cl into the cell structure. Such treatments produce an enhancement in the initial device efficiencies. However, there has been concern that Cu and Cl at the CdTe-CdS interface may have an effect on the stability of the device. In the unstressed state, CdTe devices are known to have significant amounts of both Cu and Cl at the interface. In addition, Cu placed at the back contact has been proven to diffuse toward the interface as the device is stressed, increasing the amount of Cu at the junction. There is thought to be a correlation between the amounts of Cu diffused to the interface from the back-contact and the resulting observed degradation in performance, the origin of which has yet to be determined. We have begun experiments studying how direct incorporation of Cu and Cl at the interface affects device performance. In this study, we detail the correlation observed between CuCl vapor-deposited at the CdS/CdTe interface prior to CdTe deposition with resulting device performance. X-ray diffraction, x-ray photoelectron spectroscopy, SIMS, Raman spectroscopy and optical absorption techniques are used to characterize the resulting change in the CdS films as a result of incorporating Cu and Cl. It is shown that at treatments below 250°C a CuCl layer forms on the surface of the CdS. Above 250°C, additional compounds begin to appear such as Cu@sub x@S. Absorption data shows that the band edge of the CdS shifts from 2.61eV down to 2.41eV as the CuCl treatment temperature increases. Devices are then made from the treated CdS films and their J-V data compared with data from both untreated controls and degraded devices. Device performance results are then correlated with the amounts of CuCl deposited at the interface.

3:00pm **TF2-WeA4 Cu Diffusion from Back Contacts in CdS/CdTe PV Devices***, **S.E. Asher**, T.A. Gessert, C. Narayanswamy, D. Albin, R. Dhere,* National Renewable Energy Laboratory*; C. Ferekides,* University of South Florida*; M.R. Young,* National Renewable Energy Laboratory

It has been shown that for nearly all polycrystalline CdS/CdTe photovoltaic (PV) devices, the presence of Cu within the back contact is linked to both significant improvements in device performance as well as potential long-term device instability. There have been several studies of Cu distributions in devices performed using secondary ion mass spectrometry (SIMS), and in most depth profiles the Cu appears to segregate to the CdS layer in the device. Taken with the knowledge that grain sizes in the CdS are smaller, this has suggested that Cu is distributed primarily along grain boundaries. However, artifacts in the SIMS measurements may distort the profile and lead to erroneous conclusions if differences in sputter rate and corrections for ion yield are not made. In this study we use ion implanted standards of Cu into CdTe and CdS to quantify the levels of Cu in CdTe/CdS devices before, and after, one of three different back contact processes are applied. Our results show that while Cu does increase in the CdS layer of the device, the level prior to contacting is usually lower than in the CdTe. We also show that the CdCl@sub2@ process typically performed prior to application of the back contact can introduce signicant levels of Cu into the device. Device characteristics are also compared with the SIMS diffusion profiles.

3:40pm **TF2-WeA6 Electron Cyclotron Resonance Plasma Sputtering Deposition of Highly Textured LiNbO@sub 3@ Thin Films on Si Substrates and Their Electro-Optic Properties***, **H. Akazawa**, S. Masaru,* NTT Microsystem Integration Laboratories, Japan

*Electron cyclotron resonance plasma sputtering provides high-quality thin film growth in the spontaneously generated electric field as well as the plasma stream, which carries kinetic energies of 10-30 eV. This technique has been used to grow textured LiNbO@sub 3@ (LN) thin films on Si substrates with a LiNbO@sub 3@ target. Smooth, crack-free films more than 1.2 µmm thick, were obtained. The x-ray diffraction pattern of films grown under optimized conditions revealed a single LiNbO@sub 3@ phase with its direction preferentially oriented along the substrate normal. The LiNb@sub 3@O@sub 8@ phase developed under excess O@sub 2@ gas ambient because of the enhanced re-evaporation of Li@sub 2@O molecules. ICP atomic emission spectroscopy confirmed that the composition is mainly affected by the oxygen partial pressure, but little affected by the growth temperature. Cross-sectional TEM images revealed columnar grains extending from the interface, and the volume fraction of the c-axis oriented domains was about 30%. Double inter-layers, 3-5 nm thick, (possibly a-SiO@sub 2@ and a-LN) were seen between the crystalline LN film and the Si substrate. The refractive index of the LN film was similar to the value of bulk crystal, indicating that the film was densely packed. The current-voltage characteristic was reversible up to the electric coercive force of bulk LN without destroying the insulating property, and the resistance was of the order of 10@super -11@ @ohm@cm below the applied voltage of 2 eV. The electro-optic effect was investigated by spectroscopic ellipsometry. We could detect a @delta@n value in the 10@super -4@ range, which corresponds to 30-20% of the Pockels constant of bulk crystal.

4:00pm **TF2-WeA7 Infrared Electroluminescent Zinc Sulfide: Rare Earth Doped Thin Film Devices***, **A.S. Kale**, W. Glass, N. Shepherd, M.R. Davidson, P.H. Holloway,* University of Florida

ZnS doped rare earth fluoride thin films have been fabricated by RF magnetron sputtering in the conventional metal-insulator-semiconductor-metal electroluminescence (EL) configuration as a novel source for IR radiation. These ACTFEL (alternating current thin film electroluminescent) devices have promise for applications in fiber optic communication and industrial gas sensors as well as consumer electronic devices. ZnS thin films, typically 1µm thick, doped with ErF@sub 3@ and NdF@sub 3@ have been studied for their IR versus visible emissions. Electroluminescence has been investigated after different annealing temperatures (as deposited to 475°C) at a fixed time (60min) to study the influence of annealing on the IR brightness. An extremely sharp temperature dependence has been found in the IR emission, where anneals at 425°C sharply increase the emission intensity, while temperatures 25°C higher or lower result in sharply attenuated emission. The origin of this effect will be discussed. Emission spectra and efficiency from 0.35 to 1.55µm will be reported. For Er doped films, there is no shift in 4f-4f emission wavelengths versus annealing temperature, but shifts of up to 10 nm are seen for Nd doped ZnS thin films. This shift in wavelengths in Nd has been attributed to crystal field effects on the mixed 5d-4f as well as the 4f-4f transitions. Room as well as low temperature time resolved decay measurements are presented to compare the nature of specific energy transitions and energy transfer mechanisms as function of device temperature.

4:40pm **TF2-WeA9 Dual-Color UV/IR Photodiodes Based on AlGaN Grown on Si and SOS for Advanced Fire/Flame Detectors**, ***D. Starikov***, *C. Boney, N. Medelci, R. Pillai, A. Bensaoula,* University of Houston

Rugged and reliable fire/flame detector arrays can be developed through integration of mature Si-based photodetectors with the newer UV wide band gap semiconductor photodetector technology. A GaN/InGaN/GaN double heterostructure grown on Si is photosensitive in the range from near UV to near IR. The UV range provided by the nitride layers (235-365 nm) is extended into visible and IR regions (365-1100 nm) by a Si p-n junction formed during the growth of the AlN buffer on the Si substrate. Schottky barrier photodiodes based on AlGaN layers grown on Si (detector 1) and sapphire (detector 2) can further extend the UV range of the above described structure and will be the focus of this paper. The AlGaN structures were grown using RFMBE and were processed by Cl-based reactive ion etching in order to expose each layer in the multilayer structure, deposit ohmic and Schottky barrier contacts, and investigate the electrical and photovoltaic properties of each interface. The results from detector 1 indicate high responsivity in short UV wavelengths down to 275nm, confirming the 35% Al concentration as determined by independent transmittance measurements, and in 550nm and longer range. Measurements from the detector 2 show a response limited to the UV range with a cutoff of between 290nm and 300nm indicative of an aver age Al content of ~25%. The above results are currently being implemented in the development of UV/IR sensitive tandem structures fabricated on silicon-on-sapphire (SOS) substrates. Results on the SOS-based photodetector fabrication, testing, as well as optimization of the UV to IR ratio by modeling of the component pixel structure will be presented.

5:00pm **TF2-WeA10 Gadolinium Doped Yttrium Oxide Thin Films Deposited by Radio-frequency Magnetron Sputtering; Film Quality and Cathodoluminescence Properties**, ***J.D. Fowlkes***, *P.D. Rack, Y. Deng,* The University of Tennessee, Knoxville; *J.M. Fitz-Gerald, R.K. Bansal,* The University of Virginia

Miniaturized ultraviolet (250 - 350 nm) emitting solid - state sources are required as components for proposed device structures such as non-line-of-sight communication transceivers and receivers and bioparticle detection units. Rare - earth doped, yttrium oxide thin films emit ultraviolet light over this proposed frequency range. Yttrium oxide thin films were deposited by radio - frequency magnetron sputtering in a reactive oxygen atmosphere. The films were deposited on Si (001) substrates and were polycrystalline with a preferred (222) pole orientation along the substrate normal that became more prominent at high temperature. In addition to texture, crystal size and crystal quality were determined, using x-ray diffraction, for a host of sputtering conditions. Yttrium oxide was doped with gadolinium which emits in the ultraviolet via an interband 4f-4f transition. The films emit at 314 - 315 nm with optimum intensity at ~ 10 at% Gd. Correlations have been made between thin film orientation, crystallite size, residual stress, and cathodoluminescence (CL) studies. CL excitation was optimized per sample by proper voltage, current, and temperature selection. In addition, CL data will be presented that reflects the characteristics of the Gd optical transition. Specific information regarding the phonon assisted transition and thermal and concentration quenching will be discussed.

Thin Films
Room 329 - Session TF-ThM
Modeling & Fundamentals in Thin Film Deposition

Moderator: M.C.M. van de Sanden, Eindhoven University of Technology, The Netherlands

8:20am **TF-ThM1 Mechanisms and Energetics of H Insertion into Si-Si Bonds in Hydrogenated Amorphous Silicon Films***, S. Sriraman,* ***M.S. Valipa***[1]*, E.S. Aydil,* University of California, Santa Barbara*; D. Maroudas,* University of Massachusetts

Understanding the mechanism of chemically-induced disorder-to-order structural transitions is relevant to the synthesis of various technologically important materials, including nanocrystalline silicon thin films used in the fabrication of solar cells and flat panel displays. Thin hydrogenated amorphous silicon (a-Si:H) films crystallize at low temperatures when exposed to H atoms from an H@sub 2@ plasma, or when the SiH@sub 4@ feed gas is heavily diluted with H@sub 2@. Recently, we showed that H-induced crystallization is mediated by insertion of H atoms into strained Si-Si bonds as the H atoms diffuse through the a-Si:H film.@footnote 1@ This presentation focuses on analyses of the mechanisms and energetics of various pathways for H insertion into Si-Si bonds in a-Si:H. These pathways are generated through molecular-dynamics simulations of repeated H atom impingement on a-Si:H films, grown computationally on crystalline Si (c-Si) substrates. Prior to insertion, the diffusing H atom bonds to one of the Si atoms that form the original Si-Si bond; upon insertion, an intermediate bridging configuration forms where the H atom is bonded to both Si atoms. After the H atom leaves the bridging configuration, the Si-Si bond may be further strained or broken, or it may relax to restore the Si-Si bond closer to the equilibrium c-Si bond length. A diffusing H atom can also occupy the bond-centered (BC) location between two Si atoms that are not bonded together. However, after the H atom diffuses away from this BC location, the two Si atoms form a Si-Si bond. The activation energy barriers are calculated for the H insertion reactions; the resulting distribution of activation energies is correlated with the Si-Si bond strain. The role of these H-insertion reactions in the structural relaxation of the a-Si:H network that results in disorder-to-order transitions is discussed. @FootnoteText@@footnote 1@ S. Sriraman, S. Agarwal, E. S. Aydil, and D. Maroudas, Nature 418, 62-65 (2002).

8:40am **TF-ThM2 Effect of Crosshatch Formation on the Kinetics of Si@sub 1-x@Ge@sub x@ Growth on Si(001) from Hydride Precursors***,* ***T. Spila****,* University of Illinois*; P. Desjardins,* École Polytechnique de Montréal, Canada*; J. D'Arcy-Gall, R.D. Twesten, J.E. Greene,* University of Illinois

Compressively-strained Si@sub 0.7@Ge@sub 0.3@ layers were grown on Si(001) by gas-source molecular beam epitaxy from Ge@sub 2@H@sub 6@/Si@sub 2@H@sub 6@ mixtures at 450 °C. The combination of the relatively low growth temperature and high steady-state hydrogen surface coverage, @theta@@sub H@ = 0.52 ML, suppresses strain-induced roughening and provides extremely flat surfaces with root mean square widths w < 1.5 Å for fully-coherent layers. These samples were used to probe mechanisms controlling misfit-dislocation-induced surface roughening (i.e., crosshatch) along 90°-rotated directions. For film thicknesses t just larger than the critical value for misfit dislocation formation, t@sub c@ = 1000 Å, the surface roughness (w = 3.1 Å at t = 1350 Å) is dominated by single- and multiple-atomic-height steps generated by the motion of threading dislocations associated with the interfacial misfits. The surface steps are preferential H desorption sites and the increased total step length results in a decrease in @theta@@sub H@ on terraces as well as at step edges. The latter effect allows higher adatom crossing probabilities at ascending steps leading to the formation of periodic ridges (w = 27 Å at t = 4400 Å) in response to local strain fields associated with the misfit dislocation clusters. Simultaneously, the decrease in @theta@@sub H@ on terraces strongly affects film growth kinetics as deposition rates increase from 10 Å min@super -1@ with t < t@sub c@ to 60 Å min@super -1@ with t = 1400-4400 Å. Overall, for films with strain relaxation R < 5%, crosshatch is due to surface steps that result from multiple misfit dislocations on single glide planes, but for R = 22-78%, crosshatch becomes dominated by local strain-induced roughening and leads to periodic ridge formation.

9:00am **TF-ThM3 Initial Growth of High Rate Deposited Silicon Thin Films Studied by In-situ Spectroscopic Ellipsometry and Attenuated Total Reflection Infrared Spectroscopy***,* ***P.J. Van den Oever****, I.J. Houston, J. Hong, M.C.M. van de Sanden, W.M.M. Kessels,* Eindhoven University of Technology, The Netherlands

The trend towards high rate deposition in thin film research - driven by industrial demands - raises several issues in order to maintain good film properties. One particular issue in this respect is the control of the nucleation and initial growth in terms of film density and surface roughness. Therefore, we have investigated the growth of silicon-based films at growth rates up to 10 nm/s as deposited by the expanding thermal plasma technique. The time evolution of the dielectric functions, bulk thickness and surface roughness has been obtained from real time spectroscopic ellipsometry measurements and the surface roughness evolution has been compared to atomic force microscopy data. Attenuated total reflection infrared spectroscopy has been employed to obtain the hydrogen depth profile of the films. For hydrogenated amorphous silicon, the film growth is found to be homogeneous after nucleation. Hydrogenated microcrystalline silicon on the other hand shows a nucleation region of crystallites, which is followed by columnar film growth after the crystallites have reached coalescence. This is supported by the hydrogen depth profile where the nucleation and presence of the crystallites is accompanied by the emergence of surface-like bonded hydrogen that can be attributed to hydrogen passivating the grain boundaries of the crystallites. For silicon nitride films a clear difference has been observed in the surface roughness evolution between silicon-rich and nitrogen-rich films as deposited under different plasma conditions. This difference can be attributed to different growth modes that are determined by the surface free energy and the specific surface reactions taking place during film growth.

9:20am **TF-ThM4 Sn-mediated Ge/Ge(001) Growth by Low-temperature Molecular Beam Epitaxy: Effects on Surface Roughening and Epitaxial Thickness***,* ***K.A. Bratland****, Y.L. Foo, T. Spila,* University of Illinois*; P. Desjardins,* École Polytechnique de Montréal, Canada*; J.E. Greene,* University of Illinois

Fully-strained single-crystal metastable Ge@sub 1-x@Sn@sub x@ layers were grown on Ge(001) in order to probe the role of dopant and dilute alloy concentrations (C@sub Sn@ = 1x10@super 18@ cm@super -3@ to 6 at%) on surface roughening pathways leading to epitaxial breakdown during low-temperature molecular beam epitaxy (LT-MBE) of compressively strained films. The growth temperature was chosen to be 155 °C due to limited epitaxial thicknesses at lower temperatures and significant Sn surface segregation at higher temperatures. The addition of Sn during Ge(001) LT-MBE was found to give rise to two competing effects. At very low concentrations (x < 0.02), the dominant effect is a Sn-induced enhancement in both the Ge adatom mobility and the rate of interlayer mass transport, thereby suppressing kinetic roughening and resulting in significant enhancements in the critical thickness h@sub 1@ for the onset of epitaxial breakdown. With x = 0.010, for example, the surface width w normalized to h@sub 1@, which is 2.6x10@super -3@ for pure Ge(001) layers, decreases to 1.5x10@super -3@ while h@sub 1@ increases from 7700 Å to 1.40 µm. Furthermore, layers which are fully epitaxial to thicknesses h > 1.9 µm are obtained with the incorporation of Sn in concentrations ranging from 1x10@super 18@ cm@super -3@ to 1.0 at%. However, for x @>=@ 0.02, strain-induced roughening overcomes the Sn-induced surface smoothening effects and gives rise to a rapid decrease in h@sub 1@, which ranges from 5700 Å with x = 0.029 to 2350 Å with x = 0.061.

9:40am **TF-ThM5 Understanding Radical-Surface Interactions in the Plasma-Assisted Deposition of Amorphous Hydrogenated Silicon***,* ***S. Agarwal****, S. Sriraman,* Univ. of California, Santa Barbara*; A. Takano,* Fuji Electric Corp. R&D, Ltd., Japan*; M.C.M. van de Sanden,* Eindhoven Univ. of Tech., The Netherlands*; D. Maroudas,* Univ. of Massachusetts - Amherst*; E.S. Aydil,* Univ. of California, Santa Barbara **INVITED**

Hydrogenated amorphous silicon (a-Si:H) thin films grown by plasma-assisted deposition from silane-containing discharges are widely used in photovoltaic, imaging, and display devices. During plasma-assisted deposition, reactive radicals present in the discharge such as SiH@sub x@ (x = 1,2,3) and H impinge onto the substrate surface leading to film growth and H incorporation. The properties of the deposited films depend on the various reactions of radicals present in the discharge with the growth surface and, therefore, a fundamental understanding of such interactions is required for improving a-Si:H deposition techniques. We have studied

[1] **TFD Student Award Winner**

radical-surface interactions in the deposition of a-Si:H thin-films through synergic use of experimental diagnostics such as in situ attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy and molecular-dynamics (MD) simulations based on the Tersoff's potential for Si. We have identified the various reactions of H and SiH@sub 3@ with a-Si:H films and determined their mechanisms and energetics. Surface H is removed by abstraction through the Eley-Rideal mechanism. Silicon is incorporated into the growing film through radical attachment to surface dangling bonds, as well as radical insertion into Si-Si surface bonds including dissociative adsorption reactions. In addition, reactions that involve radical migration on the surface are identified and analyzed. Finally, the H-induced crystallization of a-Si:H films during their post-deposition treatment by an H@sub 2@ plasma is addressed. The MD simulations show that H atoms diffuse into the a-Si:H film and insert into strained Si-Si bonds to form intermediate bridging and bond-centered H (Si-H-Si) configurations mediating the structural relaxation of the amorphous Si matrix leading to the disorder-to-order transition. The existence of bond-centered H(D) in a-Si:H films exposed to H(D) atoms was verified experimentally through IR spectroscopy.

11:00am **TF-ThM9 From Ab-initio Modeling to Experimental Thin Film Synthesis of a Novel Boron-oxygen-yttrium Phase***, **D. Music**, V. Chirita,* Linköping University, Sweden*; J.M. Schneider,* RWTH Aachen, Germany*; U. Helmersson,* Linköping University, Sweden

The B-O based material system is a promising candidate for a wide range of tribological applications. For example, crystalline boron suboxide has been reported to have the elastic modulus of 470 GPa. However, typical requirements to form crystalline boron suboxide include high pressure (5-7 GPa) and temperature (>2000 °C), and are very difficult to achieve with standard thin film synthesis techniques such as reactive sputtering. In this work, we use ab-initio calculations to theoretically design and then experimentally grow polycrystalline boron suboxide based films. A new crystalline boron-oxygen-yttrium (BOY) phase is obtained by alloying with Y. The essential element in the modeling is Y substituting for O in the boron suboxide structure with Y/B and O/B ratios of 0.07. The overall effect of electron doping, induced by the Y substitution, is to shorten the chemical bonds in boron suboxide. This renders the formation of the BOY phase characterized by a 45% volume reduction and consequently a 23% increase in bulk modulus (from 235 to 289 GPa). The calculations predict that the BOY phase is 0.36 eV/atom more stable than crystalline boron suboxide and experiments confirm the formation of crystalline thin films. The BOY phase was synthesized with reactive RF magnetron sputtering and identified with x-ray and selected area electron diffraction. Films with Y/B ratios ranging from 0.10 to 0.32, as determined via elastic recoil detection analysis, were grown over a wide range of temperatures (300-600 °C) and found to withstand 1000 °C. Details of the electronic structure of this new phase will also be presented.

11:20am **TF-ThM10 Low Temperature Nitride and Oxide Thin Film Growth using an Energetic Atom Source***, **A.H. Mueller**, E.A. Akhadov, M.A. Hoffbauer,* Los Alamos National Laboratory

Depositing one or more metals on a substrate exposed to an energetic neutral atom beam allows for the growth of high quality oxide and nitride films at low temperatures. The unique surface chemistry afforded by the exposure of the substrate to oxygen or nitrogen atoms possessing kinetic energies between 1 and 5 eV allows the deposition of dielectric and semiconducting films on materials that would undergo thermal decomposition under the conditions necessary for conventional thin film growth (e.g. CVD, sputtering, laser ablation, etc.). This low temperature epitaxy technique, called Energetic Neutral Atom Beam Lithography/ Epitaxy (ENABLE), permits the growth of semiconducting nitride and dielectric oxide films on substrate surfaces patterned by an organic polymer, as well as substrates consisting entirely of low meting point materials. The crystallinity, optical and electrical properties of ENABLE grown films have been examined using numerous techniques including XRD, TEM, photoluminescence spectroscopy, and four point probe resistance measurements. Results of film deposition on various substrate materials, including patterned film deposition, will be discussed.

11:40am **TF-ThM11 Physical Self-assembly and the Nucleation of 3D Nanostructures by Oblique Angle Deposition@footnote 1@***, **T. Karabacak**, G.-C. Wang, T.-M. Lu,* Rensselaer Polytechnic Institute

Thin film growth front morphology formed by physical vapor deposition is controlled by many factors including surface diffusion and shadowing effects. Instabilities can occur if shadowing is more dominant compared to other surface effects and can lead to many diverse physically self-assembled 3D nano-size structures@footnote 2,3@ that are otherwise difficult to produce by lithographical techniques. The unique geometrical shapes from a large variety of materials suggest that shadowing effects can be used as an efficient tool to understand and control the growth morphologies by many traditional deposition techniques. In this paper, we explore the fundamental nucleation and growth mechanisms of these structures. Monte Carlo simulations were carried out to predict the island density, island size distribution, and island-island correlation during the initial stages of growth. The results are compared to that obtained by the oblique angle sputter deposited W films imaged by atomic force microscopy and scanning electron microscopy. Isolated islands with quasi-periodic distribution are formed as a natural consequence of the shadowing effect. Isolated columnar structures are shown to grow on these islands and the width w of the columns is predicted to grow as a function of time t in the form of a power law, w ~ t@super p@, where the exponent p is between 0.3 and 0.5. The predicted p is consistent with the experimentally determined exponent values for growth of column widths from a variety of materials such as W, Co, Cu, and Si. @FootnoteText@ @footnote 1@ Work supported by NSF@footnote 2@ Y.-P. Zhao, D.-X. Ye, G.-C. Wang, and T.-M. Lu, "Novel nano-column and nano-flower arrays by glancing angle deposition", Nano Letters 2, 351-354 (2002). @footnote 3@ K. Robbie, M. J. Brett, and A. Lakhtakia, "Chiral sculptured thin films," Nature 384, 616-616 (1996).

Thursday Afternoon, November 6, 2003

Thin Films
Room 329 - Session TF-ThA
In-Situ / Ex-Situ & Real-Time Monitoring

Moderator: R.A. Lukaszew, University of Toledo

2:00pm **TF-ThA1 Thin-film Cavity Ringdown Spectroscopy (tf-CRDS) for Ultra-sensitive and Direct Detection of Defect-related Absorptions in a-Si:H Thin Films***, **I.M.P. Aarts**[1], B. Hoex, A.H.M. Smets, R. Engeln,* Eindhoven University of Technology, The Netherlands*; M. Nesládek,* Limburgs Universitair Centrum, Belgium*; W.M.M. Kessels, M.C.M. van de Sanden,* Eindhoven University of Technology, The Netherlands

We have explored the applicability of cavity ringdown spectroscopy with respect to the measurement of small, defect-related absorptions in thin films. First of all, the validity of the method has been confirmed by rigorous studies on the issues that arise when placing an optical transparent substrate inside the high-finesse optical cavity: it has been found that the stability of the cavity, the build-up time of the electromagnetic field inside the cavity as well as the changes in output signal are not significantly affected by the substrate. Furthermore, the losses caused by surface scattering induced by the sample's surface roughness have been estimated employing surface morphology data obtained from atomic force microscopy. It is shown that surface scattering will eventually limit the absorption sensitivity of the technique, which is as good as 10@super -7@ per pass. Subsequently, as a proof-of-principle, measurements have been performed on several samples of hydrogenated amorphous silicon (a-Si:H) thin films in a broad spectral range (0.7 - 1.7 eV) using an optical parametric oscillator laser. The absorption values of the a-Si:H films of various thickness (5 - 1000 nm) have been corrected for interference effects that have been modeled in a straightforward but complete manner. The optical absorption spectra obtained revealed good agreement with conventional transmission-reflection and photothermal deflection spectroscopy (PDS) measurements and the spatial distribution as well as the bulk and surface defect densities in the a-Si:H thin film have been determined. From the results, it can be concluded that tf-CRDS is an ultra-sensitive absorption technique that does not require any calibration. The technique is therefore very promising for a broad range of thin film research fields and currently experiments are carried out to detect ultra-low doping levels of rare earth metals in silicon and to apply the technique real time during film growth of a-Si:H.

2:20pm **TF-ThA2 Quasi-Real Time in-situ FT-IR Spectroscopy of Doped and Undoped SiO@sub 2@ Deposition from TEOS / Ozone Chemistry***, J.E. Crowell, **L.D. Flores**,* University of California, San Diego

Application of quasi- real time in-situ infrared spectroscopy to the chemical boundary layer (CBL) region formed during atmospheric pressure chemical vapor deposition is investigated for silicate glass deposition from TEOS / Ozone, and upon addition of phosphorus and boron dopants. The CBL-FT-IR methodology provides direct chemical measurement of the deposition intermediates formed upon dopant addition allowing for measurement of both film forming precursors and film formation processes. Similarly, CBL difference spectroscopy (CBL-DS) provides a sensitive measurement of the time needed for attainment of steady state reactor conditions after introduction or switching of reagents. Partial least squares Beer's law quantitative methods applied to the isolated form of ethoxysilanol (3737 cm@sup -1@) show that its concentration is reduced by a factor of 2 during growth of PSG under steady state conditions. We have developed a PLS methodology to quantitate reactive chemical mixtures of TEOS and ozone and found the chemical kinetics to be independent of added phosphite (i.e. [k@sub TEOS@/k@sub OZONE@]@sub PSG@ ~ [k@sub TEOS@/k@sub OZONE@]@sub USG@). Considering the ca. 2.8-fold enhancement in deposition rate observed upon phosphorus addition along with the measured reduction in gas phase isolated silanol groups illustrates that gas phase ethoxysilanol species are the main film deposition intermediates in equilibrium with SiO@sub 2@ film growth. This result shows that real time CBL-DS is a powerful methodology to determine the chemical mechanisms of film growth as well as the associated chemical kinetics of gas phase processes responsible for their growth. Spectroscopic differences and comparisons for PSG, BSG, and BPSG film growth and the associated intermediates will additionally be made.

[1] **TFD Student Award Winner**

2:40pm **TF-ThA3 Numerical Analysis of the Three-phase Problem in Optical Diagnostics***, **K.F. Flock**, D.E. Aspnes,* North Carolina State University

One of the major unsolved problems in optical diagnostics is the practical simultaneous determination of n, k, and t, i.e., the real and imaginary parts of the complex refractive index and the thickness, respectively, of a depositing film, ideally at the monolayer or near-monolayer level. This capability is particularly important for purely sample-driven feedback control of deposition processes such as OMCVD. For very thin layers analysis can be done in principle within the three-phase (substrate/overlayer/ambient) model, since the underlying substrate, no matter how complicated, can be represented approximately as a pseudodielectric function and the material deposited between measurements can be considered uniform in composition. Current optical diagnostic tools, such as our PDA-based polarimeter, return three pieces of information, i.e., the p- or s-polarized reflectance and the phase and amplitude of their ratio, and hence are well suited for this approach. Wavelength-by-wavelength analysis of electrochemical modulated-reflectance data, a related application, has been done previously in the three-phase model with marginal results. Here, we use a simple analytic approach to investigate correlations among n, k, and t to gain better insight into the nature of these solutions. We find that the correlation among the three parameters would be exact if the power reflectance were an analytic function. This explains the high sensitivity to experimental uncertainty, which in wavelength-by-wavelength applications would require accuracies of the order of 1 part in 10@super 6@ for consistent results. We present a method that circumvents this difficulty by taking advantage of spectral dependences. Applications discussed include the determination of n, k, and t for sub-nm-scale layers of Ga and AlAs on GaAs.

3:00pm **TF-ThA4 Mapping Epitaxial Interfaces with Ultrabright X-rays***, **R. Clarke**,* University of Michigan, Ann Arbor **INVITED**

A new direct structure determination technique, Coherent Bragg Rod Analysis (COBRA), has been developed@footnote 1@ that reveals the atomic structure of epitaxial thin films and interfaces with sub-Angstrom resolution. The measurements take advantage of the high brilliance of x-ray synchrotron radiation from undulator beam lines at the Advanced Photon Source, Argonne National Laboratory. In this presentation we will describe the capability of COBRA to reveal subtle details of the interface structure that cannot be accessed by existing structural methods. The method will be illustrated by several examples drawn from our recent work on epitaxial oxide films, including perovskite ferroelectric heterostructures. This work is carried out in collaboration with Y. Yacoby (Hebrew University, Jerusalem), R. Pindak (NSLS), and E. Stern (University of Washington) and is funded by the U.S. Department of Energy, Basic Energy Sciences, and by FOCUS, a National Science Foundation Frontiers of Physics Center. @FootnoteText@ @footnote 1@ 1. Direct determination of epitaxial interface structure: Gd2O3 passivation of GaAs, Y. Yacoby, E.Stern, J. Cross, D. Brewe, R. Pindak, D. Walko, E. Dufresne and R. Clarke, Nature Materials 1, 99-101 (2002).

3:40pm **TF-ThA6 Real Time X-ray Monitoring of Ta Film Thickness, Phase, and Texture Evolution during Sputter Deposition***, **D. Windover**,* Rensselaer Polytechnic Institute*; S.L. Lee,* ARDEC, Benet Laboratories*; T.-M. Lu,* Rensselaer Polytechnic Institute

This work focuses on X-ray reflectivity and diffraction techniques for real time monitoring of thin film deposition inside a sputtering system. An X-ray transparent, beryllium, cylindrical chamber was constructed to allow for diffraction and reflection from the multiple geometries necessary for X-ray characterization methods. A magnetron sputter head with varying target-sample distances was used as the deposition source. In this study, tantalum was deposited on silicon substrates. Fast X-ray diffraction and texture information was collected using a position sensitive area detector. Fast X-ray reflectometry was collected using an energy dispersive silicon detector. Examples of thickness, phase, and texture monitoring are presented. We conclude with a discussion on the advantages and potential limitations of the characterization approaches and their applicability to real time monitoring in deposition systems.

4:00pm **TF-ThA7 Epitaxial Growth of Nanostructured Metal/Metal Oxide Thin Films by Ultrahigh Vacuum In-situ TEM***, **M. Yeadon**,* IMRE, Singapore*; J. Yu,* National University of Singapore, Singapore*; W. Tian, H.P. Sun, X.Q. Pan,* University of Michigan*; C.B. Boothroyd,* IMRE, Singapore*; R.A. Lukaszew,* University of Toledo*; R. Clarke,* University of Michigan, Ann Arbor **INVITED**

The structure and properties of metal: metal oxide systems are of substantial importance in applications ranging from magnetic storage and

spintronic devices to supported catalysts. In some metal/oxide systems it is possible to achieve epitaxial growth and the properties of the films may strongly depend on the interfacial structure as well as their growth mode. Using a modified ultrahigh vacuum transmission electron microscope (the MERLION system), we have investigated the nucleation and growth of Ni thin films on electron transparent metal oxide substrates. The system is equipped with solid source electron beam evaporators together with gas injection capability, all within the polepiece of the electron microscope which has a base pressure of 1.5x10@super -10@Torr. The talk will focus on the early stages of nucleation and growth. Real-time video recordings of the observed microstructural evolution will be presented. Some results from related in-situ experiments involving the growth of other thin films and nanostructured materials will also be presented.

4:40pm **TF-ThA9 Growth of Epitaxial Two-dimensional Layers of Indium on Si(100) by Femtosecond Pulsed Laser Deposition***, **M.A. Hafez**, M.S. Hegazy, H.E. Elsayed-Ali,* Old Dominion University

The growth of high quality epitaxial metal films on Si is of interest to device technology. Pulsed laser deposition (PLD) produces energetic species, which result in the increase of the sticking coefficients and adatom surface mobility enhancing epitaxial growth. Compared to longer laser pulses, femtosecond (fs) laser pulses significantly alter the processes associated with ablation. Epitaxial growth of thin films by fsPLD has been previously reported. Heteroepitaxial growth of indium on Si(100)-(2x1) substrates is performed under ultrahigh vacuum (base pressure low 10@super -9@ Torr) with a Ti:sapphire laser (130 fs) at wavelength of 800 nm. Reflection high-energy electron diffraction (RHEED) is used during the deposition to study the growth dynamics and the surface structure of the grown films. Morphology of the indium films is examined by ex-situ atomic force microscopy (AFM). At a substrate temperature of ~145 °C, indium was initially found to grow two-dimensionally, and then three-dimensional islands were formed. Epitaxial indium thin films are found to grow at a deposition temperature of ~400 °C, which have a 3x4 structure as observed from the RHEED pattern. The RHEED analyses and intensity oscillations show that indium films grow with two-dimensional layers and the AFM reveals two-dimensional nucleation islands. Growth modes and nucleation of indium films at different deposition conditions are discussed.

5:00pm **TF-ThA10 Real-time Observation of Initial Stages of Copper Film Growth on Silicon Oxide using Reflection High-energy Electron Diffraction***, **J.T. Drotar**,* Rensselaer Polytechnic Institute

We have studied, in real time, the evolution of a thin (less than 200 Å) copper film deposited onto an oxidized silicon surface using reflection high-energy electron diffraction (RHEED). We show that quantitative measurements of island size and shape as functions of time are possible and the results are presented. While the film texture is initially random, texture competition leads to an absence of the low energy (111) and (200) oriented grains for later times. It is also found that the film surface is composed of facets that increase in size with time. This behavior is explained in terms of facet coalescence.

Thin Films
Room 329 - Session TF-FrM
Mechanical Properties of Thin Films

Moderator: B.C. Holloway, College of William & Mary

8:20am **TF-FrM1 Island Coalescence Stress Created During Thin Film Growth***, **S.J. Hearne**, J.A. Floro, C. Dyck, T. Christenson,* Sandia National Laboratories*; W. Fan, S.R.J. Brueck,* University of New Mexico

For over 30 years it has been generally accepted that island coalescence during thin film growth results in a tensile stress. However, prior to this work there had been no conclusive demonstration of the fundamental physical mechanism underlying this process. Using selective lateral film growth on patterned substrates during electrodeposition of Ni films, we have obtained the first unambiguous comparison of the experimentally-measured tensile stress due to island coalescence with theoretical predictions. This technique allows for the systematic variation of island size and geometry while maintaining temporally constrains of the coalescence of Ni islands during electroplating. This avoided the complications associated with stochastic island coalescence that have plagued previous measurements obtained over the last 30 years. We found that the functional dependence of the stress on island size and dimensionality is consistent with a Hertzian contact model recently proposed by Freund and Chason.@footnote 1@ However, the absolute magnitude of the measured stress is smaller by factors of 2-4x, and reasons for the discrepancy will be discussed. This work was partially supported by the DOE Office of Basic Energy Sciences. Sandia is a multiprogram laboratory of the United States Department of Energy operated by Sandia Corporation, a Lockheed Martin Company, under contract DE-AC04-94AL85000. @FootnoteText@ @footnote 1@L.B. Freund, E. Chason, JAP 89, 4866 (2001).

8:40am **TF-FrM2 Alloying Effects on Mechanical Behavior of Thin Metal Films***, **R.P. Vinci**,* Lehigh University **INVITED**

Thin metal films such as Al, Pt and Au are often used in their pure state in microelectronics and MEMS applications. This is true despite the fact that their mechanical behavior is frequently critical for performance. When thin film alloys are employed, a specific alloy composition is often selected because it is a preexisting combination found in a particular fabrication facility, not because it is the optimum composition for the application at hand. In contrast, bulk metals are almost always alloyed for mechanical and microstructural stability, and the effects of various alloying elements are well characterized. Should a designer of microelectronics and MEMS applications need a particular set of mechanical properties (e.g., fatigue or creep resistance) there is no available equivalent understanding of alloy effects in thin film form. In this presentation, systematic experiments focused on elucidating the effects of alloying on thin film mechanical properties will be described. In particular, deviation from (or agreement with) bulk behavior will be discussed for several metal alloy systems.

9:20am **TF-FrM4 Mechanical Properties of Reacted Metal Multilayers***, **D.P. Adams**, M. Rodriguez, J.B. Kelley, T. Covert, T. Buchheit, M. Grubelich,* Sandia National Laboratories

Sputtered multilayer thin films composed of reactive material pairs are currently of interest for brazing, joining and other energetic applications. As shown extensively by Weihs et.al. several thin film material systems can be stimulated such that a rapid, self-propagating reaction occurs within a multilayer. This alloying reaction has great potential for joining, because it prevents global heating of parent materials. In this talk, we evaluate the resultant mechanical properties and microstructure of three different reacted multilayer systems. This includes Ni/Ti, Al/Pt and metal/B multilayers. Each material system was deposited by magnetron sputtering to thicknesses in excess of 1 ï•-m. First, we describe how the propagation velocity depends on multilayer design. Propagation velocities are measured by imaging the propagating reaction front using a high speed Cordin camera and strobe light assembly. Velocities in excess of 50 m/s are measured for some multilayer designs. We determine how changes in stoichiometry (specifically deviations from the targeted composition) affect velocity. Secondly, we discuss the stress in reacted films. Laser-based wafer curvature techniques are used to determine average in-plane stress in reacted films. Stresses in excess of 1 GPa have been measured for several reacted multilayers. To better understand the development of stress in these systems we have conducted x-ray diffraction to determine coefficients of thermal expansion. Films achieve extremely high temperatures during self-propagating reactions and extrinsic stress largely determines final stress state; this can greatly affect their usefulness in a braze process. Additional mechanical properties such as hardness are measured, and these are related to film microstructure probed by transmission electron microscopy.

9:40am **TF-FrM5 Intrinsic Stresses in CrN Films Deposited by Arc Ion Plating***, T. Hanabusa, **K. Kusaka**,* Tokushima University, Japan*; T. Matsue,* Niihama National College of Technology, Japan

It is well known that large compressive residual stresses of the order of GPa develop in TiN and CrN films deposited by ion plating. The residual stress in the film is the sum of thermal stress and intrinsic stress. The former is the stress originated by the difference in the thermal contraction between the substrate material and the film, whereas the latter is originated by various factors during the depositing process. The residual stress in TiN or CrN film is predominantly the intrinsic stress. The object of this study is to investigate an essential meaning of the intrinsic stress in the films. In this experiment CrN film was deposited on a stainless steel substrate. Residual stress in the CrN film was measured by X-ray diffraction. The X-ray residual stress measurement revealed that macroscopic compressive stress and microscopic stress in the film are reduced by annealing the film/substrate system. The relation between the lattice strain and the applied stress is investigated for the specimen annealed at various temperature stage.

10:20am **TF-FrM7 Characterization of TiN Films Deposited on Electroformed Nickel Substrates***, **M. Telgarsky**, M.K. Ghantasala, E.C. Harvey, Y. Wang,* Swinburne University of Technology, Australia

The properties of thin films are mainly influenced by that of their substrates viz., the structure and microstructure. This paper describes the results of our investigations in understanding the effect of structure and microstructure of the electroformed Nickel substrate on the growth and properties of deposited TiN films. Nickel substrates were electroformed using two different Nickel sulfamate baths to thicknesses of between 100 and 200 µm. The grain size of these films was estimated to be around 15 to 50 nm. The grain size of the films formed in bath 1 was much larger than grains formed in bath 2. Interestingly, the films plated in bath 1 showed (200) orientation compared to those deposited in bath 2, which exhibited (111) orientation. Further, Nickel plated in bath 1 showed much better crystallinity compared to that deposited in bath 2. After releasing from their substrates, the electroformed Nickel foils were used in turn as substrates for the deposition of TiN thin films using filtered arc deposition (FAD). All films were deposited on the Nickel substrates under the same conditions. The electroformed Nickel and the TiN films were analyzed using Rutherford Backscattering Spectroscopy (RBS), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM) and Confocal Microscopy for analysis of composition, structure, microstructure and surface profile respectively. The presence of small quantities of oxygen was detected in the electroformed Nickel from both baths. RBS analysis of the TiN films indicated that they were stoichiometric. TiN films deposited at room temperature were nano-crystalline, while those deposited at higher substrate temperatures showed the formation of improved crystalline structures having a (111) or (200) orientation. The grain size of the TiN films varied between 35 to 70 nm and showed a strong dependence on the grain size of Nickel and substrate temperature during deposition. The dependence of the TiN film properties on the Nickel substrate characteristics will be discussed in detail in this paper.

10:40am **TF-FrM8 Novel Mechanical, Electrical and Optical Properties of Al and Mg Doped Boron Thin Films***, **Y. Tian**,* Iowa State University

A new superhard and conductive (Al, Mg)-doped boron thin film with AlMgB@sub 14@ stoichiometry has been developed in this work for potential application as a hard coating on MEMS components and conductive atomic force microscope cantilevers. AlMgB@sub 14@ films were prepared on Si (100) using pulsed laser deposition at room temperature and 573 K. Transmission electron microscopy analysis reveals that the film structures are amorphous irrespective of substrate temperature during deposition. Nanoindentation tests show that hardnesses of 45 GPa and 51 GPa have been achieved in AlMgB@sub 14@ films deposited at room temperature and 573 K respectively. Except for their superhardness, AlMgB@sub 14@ films also display very low electrical resistivity and high optical absorption in a broad spectrum range, all these phenomena can be accounted for based on an electron transfer mechanism enhanced in amorphous structures.

11:00am **TF-FrM9 Synthesis of Super-Elastic Fullerene-Like Carbon Nitride Coatings by Unbalanced Reactive Magnetron Sputtering***, **J. Neidhardt,*** Linköping University, Sweden*; Zs. Czigány,* Hungarian Academy of Sciences*; L. Hultman,* Linköping University, Sweden

Carbon nitride (CN@sub x@) is an emerging material for wear-resistant coatings. Out of several characteristic structures, the so-called fullerene-like (FL) compounds are the most promising. The FL structure leads to extraordinary mechanical properties such as an extreme elasticity combined with high fracture toughness as assessed by nano indentation. Yet, FL-CN@sub x@ shows a low-to-moderate resistance to penetration. Hence, deformation energy is predominantly stored elastically and released after unload giving it the resilient character. The key for understanding its resilient character lies in a unique microstructure of bent, cross-linked and frequently intersecting nitrogen-containing graphite sheets, denoted "fullerene-like". This presentation elaborates on the growth mechanisms of FL CN@sub x@ synthesised by unbalanced reactive magnetron sputtering of graphite in a nitrogen-containing atmosphere. Preformed C@sub x@N@sub y@ (x, y < 2) species originating from the target were assigned a crucial role for the fullerene-like structure evolution. In fact, the conducted plasma analysis revealed that the majority of the film forming flux comprises multi atomic nitrogen-containing species, which are partly sputtered and also thermally emitted from the self-modified target surface. The arrival of C@sub x@N@sub y@ molecules besides carbon atoms at the substrate surface adds much complexity to the growth mechanisms. C@sub x@N@sub y@ species may act as growth templates whereas an oriented incorporation along the edges makes them more stable towards preferential chemical desorption and therefore the formation of extended and curved sheet-like structures is promoted. Also the role and extent of temperature dependent chemical desorption (sputtering) process for the FL structure evolution was investigated in order to identify the type of species incorporated.

11:20am **TF-FrM10 Hydrogenated Carbon Films and Sulfur Stabilization: Synthesis and Tribiological Characterization***, **C.A. Freyman,** Y.H. Yu, Y.W. Chung,* Northwestern University

Hydrogenated carbon films have been deposited on Si substrates and stainless steel ball bearings by magnetron sputtering. Hydrogen concentration was controlled by the percentage of hydrogen in the Ar/H@sub 2@ mixture. The friction coefficient showed a strong dependence on both the hydrogen content of the precursor gas and the relative humidity during testing. Twenty-five percent of hydrogen in the precursor gas produced the lowest frictional coefficient (0.01) at the lowest relative humidity measured (5%). Sulfur was incorporated into the film via the introduction of hydrogen sulphide in the precursor gas. Here, we attempted to use sulphur to make the surface hydrophobic and hence minimize the relative humidity effect. Hardness, friction and sensitivity towards the environment will be investigated as a function of deposition parameters.

Author Index

Bold page numbers indicate presenter

— A —
Aarts, I.M.P.: TF-ThA1, **25**
Abatemarco, T.D.: TF-MoM5, 1
Abe, M.: TF-WeP5, 12
Acosta, G.: TF-WeM3, 10
Adams, D.P.: TF-FrM4, **27**
Agarwal, S.: TF-ThM5, **23**
Aguilar, M.A.: TF-WeP24, 15
Ahn, J.Y.: TF-MoM10, 2
Akazawa, H.: TF2-WeA6, **21**
Akhadov, E.A.: TF-ThM10, 24
Albin, D.: TF2-WeA3, 21; TF2-WeA4, 21
Alkemade, P.F.A: TF-WeP32, 17
Allongue, P.: MI+TF-WeA7, **19**
Allred, D.: TF-WeM3, **10**
Anantharaman, M.R.: TF-TuM11, **7**
Anderson, J.: TF-TuA3, 8
Anglos, D.: TF-WeP20, 15
Asher, S.E.: TF2-WeA3, 21; TF2-WeA4, **21**
Asmussen, J.: TF-WeP30, 16
Aspnes, D.E.: TF-ThA3, 25
Avasthi, D.K.: TF-TuM11, 7
Aydil, E.S.: TF-ThM1, 23; TF-ThM5, 23
Aznarez, J.A.: TF-WeP24, 15
— B —
Bae, J.S.: TF-WeP15, 14; TF-WeP16, 14
Baik, H.K.: TF-WeP27, 16
Baldwin, D.: MI+TF-WeA10, 19
Bansal, R.K.: TF2-WeA10, 22
Barrell, Y.: TF-MoA9, 4
Baumann, H.: TF-WeP33, 17
Beall, J.A.: TF+MM-WeA4, 20
Bender, J.P.: TF-TuA3, 8
Benedikt, J.: PS+TF-TuM4, **5**
Bensaoula, A.: TF2-WeA9, 22
Beyer, G.: TF-MoA6, 3
Beyer, J.: TF+MM-WeA4, 20
Bitner, M.D.: TF-MoA8, 4; TF-WeP4, 12
Boney, C.: TF2-WeA9, 22
Boothroyd, C.B.: TF-ThA7, 25
Brand, O.: TF+MM-WeA1, **19**
Bratland, K.A.: TF-ThM4, **23**
Brennan, D.: TF-TuM4, 6
Bright, V.M.: TF-MoA5, 3
Brueck, S.R.J.: TF-FrM1, 27
Bruns, M.: TF-WeP33, **17**
Bryant, P.: TF-WeM6, 11
Buchheit, T.: TF-FrM4, 27
Bukowski, T.J.: TF2-WeA3, **21**
Byon, E.: TF-WeP29, **16**
— C —
Cadieu, F.J.: MI+TF-WeA6, **19**
Cai, Y.: TF-MoA4, 3
Cernanský, M.: TF-WeP20, 15
Chaker, M.: TF-WeP11, 13
Chang, K.S.: TF1-WeA9, **20**
Chen, L.: MI+TF-WeA6, 19
Chiang, H.Q.: TF-TuA3, 8
Chirita, V.: TF-ThM9, 24
Chou, H.B.: TF-WeP26, 15
Christenson, T.: TF-FrM1, 27
Chrpbak, C.: PS+TF-TuM11, 6
Chung, U.: TF-MoM10, 2
Chung, Y.W.: TF-FrM10, 28
Cionca, C.: MI+TF-WeA4, 18
Clarke, R.: MI+TF-WeA4, 18; TF-ThA4, **25**; TF-ThA7, 25
Compaan, A.D.: TF2-WeA1, **21**
Covert, T.: TF-FrM4, 27
Creatore, M.: TF-MoA9, **4**; TF-TuA5, 8
Crowell, J.E.: TF-ThA2, 25
Czigány, Zs.: TF-FrM9, 28
— D —
D'Arcy-Gall, J.: TF-ThM2, 23
Davidson, M.R.: TF2-WeA7, 21
Davis, R.: TF-WeM3, 10
Deguchi, T.: TF-WeM4, **10**
Deiker, S.: TF+MM-WeA4, 20
Delahoy, A.E.: TF-TuA2, **8**
DelRio, F.W.: TF-MoA5, 3
Deng, Y.: TF1-WeA10, **20**; TF2-WeA10, 22
Desjardins, P.: TF-ThM2, 23; TF-ThM4, 23
Devolder, T.: MI+TF-WeA7, 19
Dhere, R.: TF2-WeA4, 21
Dibbs, M.G.: TF-TuM4, **6**
Doran, A.: MI+TF-WeA9, 19
Doriese, W.B.: TF+MM-WeA4, 20
Drotar, J.T.: TF-ThA10, **26**
Dufresne, E.: MI+TF-WeA4, 18
Dyck, C.: TF-FrM1, 27
— E —
Eapen, K.C.: TF+MM-WeA3, 20
Economou, D.J.: TF-MoA10, 4
Elsayed-Ali, H.E.: TF-ThA9, 26
Engeln, R.: TF-ThA1, 25
Erlat, A.G.: TF-TuM3, 6
Exarhos, G.J.: TF-TuA1, 8
— F —
Fabreguette, F.H.: TF-MoM4, 1; TF-MoM9, **1**
Falco, L.A.: TF-WeP2, **12**
Fan, W.: TF-FrM1, 27
Ferekides, C.: TF2-WeA4, 21
Ferguson, J.D.: TF-MoM11, 2
Ferrer, S.: MI+TF-WeA5, 18
Fitz-Gerald, J.M.: TF1-WeA10, 20; TF2-WeA10, 22
Flanagan, K.W.: TF-TuM3, 6
Flock, K.F.: TF-ThA3, **25**
Flores, L.D.: TF-ThA2, **25**
Floro, J.A.: TF-FrM1, 27
Foo, Y.L.: TF-ThM4, 23
Fotakis, C.: TF-WeP20, 15
Fourcher, J.: TF-MoA10, 4
Fowlkes, J.D.: TF1-WeA10, 20; TF2-WeA10, **22**
Freyman, C.A.: TF-FrM10, **28**
Fukuda, S.: TF-WeP31, 16
Fukumoto, H.: TF-TuA6, 8; TF-WeP14, 14
Fukunaga, Y.: TF-WeP5, 12
— G —
Gaidi, M.: TF-WeP11, 13
García, E.: TF-WeP28, 16
George, E.P.: TF1-WeA8, 20
George, S.M.: TF-MoA3, 3; TF-MoA5, 3; TF-MoM11, **2**; TF-MoM4, 1; TF-MoM9, 1
Gessert, T.A.: TF2-WeA4, 21
Ghantasala, M.K.: TF-FrM7, 27
Gibson, D.G.: TF-WeM1, 10
Glass, W.: TF2-WeA7, 21
Goodwin, S.: TF-WeM6, 11
Gordon, R.G.: TF-MoM7, **1**
Grachev, S.Yu.: TF-WeP32, 17
Graham, M.E.: TF-WeP26, 15
Greene, J.E.: TF-ThM2, 23; TF-ThM4, 23
Greig, D.: MI+TF-WeA3, 18
Groenen, R.: TF-TuA5, **8**
Groner, M.D.: TF-MoA3, **3**
Grotjohn, T.A.: TF-WeP30, 16
Grubelich, M.: TF-FrM4, 27
Gundel, A.: MI+TF-WeA7, 19
Guo, S.Y.: TF-TuA2, 8
— H —
Hafez, M.A.: TF-ThA9, **26**
Hampshire, J.: TF-WeM1, 10
Hanabusa, T.: TF-FrM5, 27
Hanada, T.: TF-TuM7, 6
Hara, H.: TF-TuM7, **6**
Hara, N.: TF-WeP29, 16
Harvey, E.C.: TF-FrM7, 27
Hashimoto, K.: TF-WeP5, 12
Hayashi, Y.: TF-TuA6, 8; TF-WeP14, 14
Hearne, S.J.: TF-FrM1, **27**
Hegazy, M.S.: TF-ThA9, 26
Heller, C.: TF-TuM3, 6
Helmersson, U.: TF-ThM9, 24
Henn-Lecordier, L.: TF-MoA4, 3
Herrmann, C.F.: TF-MoA5, **3**
Hilton, G.C.: TF+MM-WeA4, 20
Hoex, B.: TF-ThA1, 25
Hoffbauer, M.A.: TF-ThM10, 24
Hoffman, R.L.: TF-TuA3, 8
Hoivik, N.D.: TF-MoA5, 3
Holloway, P.H.: TF2-WeA7, 21; TF-TuA1, 8; TF-WeP15, 14; TF-WeP16, 14
Hong, D.: TF-TuA3, 8
Hong, J.: TF-ThM3, 23
Horikawa, A.: TF-WeP21, 15
Houston, I.J.: TF-ThM3, 23
Hozumi, A.: TF-WeP3, 12
Hultman, L.: TF-FrM9, 28
Hwang, M.S.: TF-WeP19, 15
Hylton, T.: MI+TF-WeA10, 19
— I —
Ida, S.: TF-TuA8, 8
Ihara, K.: TF-WeP17, 14
Inoue, T.: TF-WeP21, **15**
Irwin, K.D.: TF+MM-WeA4, 20
— J —
Jackson, T.N.: TF-TuM1, **6**
Jacobs, T.: TF-MoA10, 4
Janssen, G.C.A.M.: TF-WeP32, 17
Jelinek, M.: TF-WeP20, **15**
Jeon, H.: TF-MoM6, 1; TF-WeP1, 12
Jeong, H.S.: TF-WeP18, 14; TF-WeP19, **15**
Jeong, J.H.: TF-WeP15, 14; TF-WeP16, **14**
Jeong, J.Y.: TF-TuA3, 8
Jimenez-Sandoval, S.: TF-WeP7, **12**
Johnson-Steigelman, H.T.: MI+TF-WeA2, 18
Jones, J.E.: TF-TuM6, **6**
Joseph Mathai, C.: TF-TuM11, 7
Jurczyk, B.: PS+TF-TuM11, **6**
— K —
Kale, A.S.: TF2-WeA7, **21**
Kameyama, T.: TF-WeP3, 12
Kamminga, J.-D.: TF-WeP32, **17**
Kanai, N.: TF-WeP5, **12**
Karabacak, T.: TF-ThM11, **24**
Karwacki, E.J.: TF-WeP4, 12
Karwacki, G.J.: TF-MoA8, 4
Kawamata, K.: TF-WeM4, 10
Kelley, J.B.: TF-FrM4, 27
Kelly, M.J.: TF-MoM5, **1**
Kessels, W.M.M.: TF-MoA9, 4; TF-ThA1, 25; TF-ThM3, 23
Keszler, D.A.: TF-TuA3, 8
Kim, C.I.: TF-WeP12, 13; TF-WeP13, 13
Kim, H.: TF-MoA1, **3**
Kim, H.S.: TF-MoM10, 2
Kim, I.W.: TF-WeP16, 14
Kim, J.G.: TF-MoM10, 2
Kim, K.C.: TF-WeP27, **16**
Kim, K.T.: TF-WeP12, 13; TF-WeP13, 13
Kim, M.G.: TF-WeP18, **14**
Kim, S.I.: TF-WeP1, 12
Kim, T.-W.: TF-TuM3, 6
Kim, Y.: TF-MoM6, 1
Kim, Y.T.: TF-WeP1, 12
Kinbara, A.: TF-WeM4, 10
Kirschner, J.: MI+TF-WeA5, 18
Kisting, S.: TF-TuM4, 6
Kita, M.: PS+TF-TuM7, 5
Klein, T.M.: TF-WeP2, 12

Author Index

Klemberg-Sapieha, J.E.: TF-WeM5, 10
Klini, A.: TF-WeP20, 15
Koga, K.: PS+TF-TuM7, 5
Koike, K.: TF-WeP31, **16**
Kondo, K.: TF-TuA6, 8
Koo, J.: TF-MoM6, 1
Koumoto, K.: TF-WeP3, 12
Kulishov, M.: TF-WeP11, 13
Kurahashi, N.: MI+TF-WeA9, 19
Kusaka, K.: TF-FrM5, **27**
Kusano, E.: TF-WeM4, 10
Kwon, S.J.: TF-WeP19, 15
Kwon, S.-K.: TF-WeP29, 16
— L —
Lacour, S.P.: TF-TuM6, 6
Langlois, O.: TF-WeP11, 13
Lannon Jr., J.: TF-WeM6, **11**
Lee, H.J.: TF-WeP19, 15
Lee, J.: TF-MoM6, **1**
Lee, J.M.: TF-WeP12, **13**
Lee, J.S.: TF-WeP16, 14
Lee, S.L.: TF-ThA6, 25
Lee, S.M.: TF-WeP27, 16
Lee, Y.C.: TF-MoA5, 3
Lei, W.: TF-MoA4, **3**
Li, N.: PS+TF-TuM5, **5**
Lim, J.W.: TF-MoM10, **2**
Liu, Y.-K.: MI+TF-WeA1, **18**
Lu, E.D.: MI+TF-WeA2, **18**
Lu, T.-M.: TF-ThA6, 25; TF-ThM11, 24
Lukas, A.S.: TF-MoA8, **4**; TF-WeP4, 12
Lukaszew, R.A.: MI+TF-WeA4, 18; TF-ThA7, 25
Lutz, H.: TF-WeP33, 17
Lyman, P.F.: MI+TF-WeA2, 18
— M —
Maeda, K.: TF-WeP6, 12
Maex, K.: TF-MoA6, 3
Malins, A.E.R.: MI+TF-WeA3, 18
Mantei, T.D.: PS+TF-TuM3, **5**; PS+TF-TuM9, 6
Margot, J.: TF-WeP11, **13**
Maroudas, D.: TF-ThM1, 23; TF-ThM5, 23
Maroun, F.: MI+TF-WeA7, 19
Martinu, L.: TF-WeM5, 10
Masaru, S.: TF2-WeA6, 21
Masse, J.-P.: TF-WeM5, 10
Matsue, T.: TF-FrM5, 27
Matsuo, K.: TF-TuA6, 8
Matthew, J.A.D.: MI+TF-WeA3, 18
McConnelee, P.A.: TF-TuM3, 6
Medelci, N.: TF2-WeA9, 22
Melendez-Lira, M.: TF-WeP7, 12
Meyerheim, H.L.: MI+TF-WeA5, **18**
Minami, T.: TF-TuA8, 8; TF-WeP17, 14; TF-WeP9, 13
Minamino, Y.: TF-TuA8, 8
Miyata, T.: TF-TuA8, **8**; TF-WeP17, 14
Moon, A.: TF-WeP30, **16**
Moon, B.K.: TF-WeP15, 14
Moon, J.T.: TF-MoM10, 2
Moriga, T.: TF-TuA6, **8**; TF-WeP14, 14
Morton, S.A.: MI+TF-WeA3, 18
Mueller, A.H.: TF-ThM10, **24**
Munford, M.L.: MI+TF-WeA7, 19
Munuera, C.: TF-WeP24, 15
Murai, K.: TF-TuA6, 8; TF-WeP14, 14
Music, D.: TF-ThM9, **24**
— N —
Nainaparampil, J.J.: TF+MM-WeA3, **20**
Nakabayashi, I.: TF-TuA6, 8; TF-WeP14, 14
Narayanswamy, C.: TF2-WeA4, 21
Neal, J.R.: MI+TF-WeA3, 18
Neidhardt, J.: TF-FrM9, **28**
Nesládek, M.: TF-ThA1, 25
Neumann, M.: PS+TF-TuM11, 6
Norris, B.J.: TF-TuA3, 8
— O —
Ogino, M.: TF-WeP23, 15
Ohashi, M.: TF-WeP21, 15
Ohdaira, T.: TF-WeP6, **12**
Ohsaki, H.: TF-WeP5, 12
Okamura, T.: TF-WeP31, 16
Oleson, J.: TF-WeM6, 11
Oliva, A.I.: TF-WeP24, 15
O'Neill, M.L.: TF-MoA8, 4; TF-WeP4, **12**
Oswald, J.: TF-WeP20, 15
Otaño, W.: TF-WeP28, 16
Owings, R.R.: TF-TuA1, **8**
— P —
Pace, C.C.: TF-WeM6, 11
Pan, X.Q.: TF-ThA7, 25
Pankow, J.: TF2-WeA3, 21
Pantojas, V.M.: TF-WeP28, **16**
Park, C.-H.: TF-TuA3, 8
Park, K.J.: TF-TuM8, **7**
Parsons, G.N.: TF-MoM5, 1; TF-TuM8, 7
Peterson, B.K.: TF-MoA8, 4; TF-WeP4, 12
Pharr, G.M.: TF1-WeA8, 20
Phillips, B.S.: TF+MM-WeA3, 20
Pillai, R.: TF2-WeA9, 22
Popescu, R.: MI+TF-WeA5, 18
Price, M.: TF-TuA3, 8
— Q —
Qi, Y.: PS+TF-TuM9, **6**
Qiu, Z.Q.: MI+TF-WeA9, 19
Quan, J.J.: MI+TF-WeA10, 19
— R —
Rack, P.D.: TF1-WeA10, 20; TF2-WeA10, 22
Ramos, N.R.: TF-WeP28, 16
Rar, A.: TF1-WeA8, **20**
Reinhard, D.K.: TF-WeP30, 16
Reintsema, C.D.: TF+MM-WeA4, 20
Ritala, M.: TF-MoM2, **1**
Robach, O.: MI+TF-WeA5, 18
Rocklein, M.N.: TF-MoM4, **1**
Rodriguez, E.: TF-WeP24, 15
Rodriguez, M.: TF-FrM4, 27
Rossnagel, S.M.: TF-MoA1, 3
Rubloff, G.W.: TF-MoA4, 3
Rudolphi, M.: TF-WeP33, 17
Ruzic, D.N.: PS+TF-TuM11, 6; PS+TF-TuM5, 5
— S —
Sacedon, J.L.: TF-WeP24, **15**
Saiki, C.: TF-WeP5, 12
Sakamoto, N.: TF-WeP21, 15
Sampei, Y.: TF-WeP21, 15
Sander, D.: MI+TF-WeA5, 18
Santoni, A.: TF-WeP20, 15
Saravanan, S.: TF-TuM11, 7
Schad, R.: MI+TF-WeA1, 18
Schaepkens, M.: TF-TuM3, **6**
Scheck, C.: MI+TF-WeA1, 18
Schmidt, J.E.: MI+TF-WeA7, 19
Schmitt, J.P.M.: PS+TF-TuM1, **5**
Schneider, J.M.: TF-ThM9, 24
Scholl, A.: MI+TF-WeA9, 19
Schuelke, T.: TF-WeP30, 16
Seddon, E.A.: MI+TF-WeA3, 18
Seidel, T.E.: TF-MoM9, 1
Seo, H.J.: TF-WeP15, 14
Seo, W.S.: TF-WeP3, 12
Seo, Y.W.: TF-WeP18, 14; TF-WeP19, 15
Shaw, J.: TF-TuM4, 6
Shen, T.-H.: MI+TF-WeA3, 18
Shepherd, N.: TF2-WeA7, 21
Shida, S.: TF-WeP21, 15
Shimakawa, T.: TF-WeP9, **13**
Shimizu, R.: TF-WeP23, **15**
Shimogaki, Y.: TF-WeP23, 15
Shioya, Y.: TF-WeP6, 12
Shirahata, N.: TF-WeP3, **12**
Shiratani, M.: PS+TF-TuM7, **5**
Shiro, T.: TF-TuM7, 6
Shiu, C.B.: TF-WeP10, **13**
Sim, H.S.: TF-WeP1, **12**
Smets, A.H.M.: TF-ThA1, 25
Solomon, S.: TF-WeM6, 11
Son, M.: TF-WeP29, 16
Son, Y.H.: TF-WeP13, **13**
Soukup, L.: TF-WeP20, 15
Spangenberg, M.: MI+TF-WeA3, 18
Specht, E.D.: TF1-WeA8, 20
Spila, T.: TF-ThM2, **23**; TF-ThM4, 23
Sriraman, S.: TF-ThM1, 23; TF-ThM5, 23
Stafford, L.: TF-WeP11, 13
Starikov, D.: TF2-WeA9, **22**
Subha, S.: MI+TF-WeA10, 19
Sugiyama, M.: TF-WeP23, 15
Sugomoto, K.: TF-WeP29, 16
Sun, H.P.: TF-ThA7, 25
Suo, Z.: TF-TuM6, 6
Suzuki, R.: TF-WeP6, 12
Szymanowski, H.: TF-WeM5, 10
— T —
Taj, S.: PS+TF-TuM11, 6
Takano, A.: TF-ThM5, 23
Takenaka, K.: PS+TF-TuM7, 5
Takeshita, M.: PS+TF-TuM7, 5
Takeuchi, I.: TF1-WeA6, **20**
Tanaka, H.: TF-WeP17, **14**; TF-WeP9, 13
Tate, J.: TF-TuA3, 8
Teer, D.G.: TF-WeM1, 10
Telgarsky, M.: TF-FrM7, **27**
Tian, W.: TF-ThA7, 25
Tian, Y.: TF-FrM8, **27**
Tobin, J.G.: MI+TF-WeA3, 18
Tominaga, K.: TF-TuA6, 8; TF-WeP14, **14**
Townsend, P.: TF-TuM4, 6
Toyoda, N.: TF-WeM2, **10**
Travaly, Y.: TF-MoA6, **3**
Twesten, R.D.: TF-ThM2, 23
— U —
Ullom, J.N.: TF+MM-WeA4, **20**
— V —
Vale, L.R.: TF+MM-WeA4, 20
Valencia, M.M.: TF-TuA3, 8
Valipa, M.S.: TF-ThM1, **23**
van de Sanden, M.C.M.: PS+TF-TuM4, 5; TF-MoA9, 4; TF-ThA1, 25; TF-ThM3, 23; TF-ThM5, 23; TF-TuA5, 8
Van den Oever, P.J.: TF-ThM3, **23**
Van Hove, M.: TF-MoA6, 3
Vargas-Lopez, E.: PS+TF-TuM11, 6
Venkatachalam, S.: TF-TuM11, 7
Vincent, J.L.: TF-MoA8, 4; TF-WeP4, 12
Vinci, R.P.: TF-FrM2, **27**
Volintiru, I.M.: TF-TuA5, 8
Vrtis, R.N.: TF-MoA8, 4; TF-WeP4, 12
— W —
Waddill, G.D.: MI+TF-WeA3, **18**
Wadley, H.N.G.: MI+TF-WeA10, 19
Wager, J.F.: TF-TuA3, **8**
Wagner, S.: TF-TuM6, 6
Walko, D.: MI+TF-WeA4, 18
Walls, J.M.: TF-WeM1, **10**
Wang, G.-C.: TF-ThM11, 24
Wang, Q.: TF-MoA10, **4**
Wang, Y.: TF-FrM7, 27
Watanabe, T.: TF-WeP5, 12
Watanabe, Y.: PS+TF-TuM7, 5
Weimer, A.W.: TF-MoM11, 2
White, B.: TF-MoA10, 4
Williams, M.: PS+TF-TuM11, 6
Windisch, C.F.: TF-TuA1, 8
Windover, D.: TF-ThA6, **25**

Author Index

Woen, R.V.: PS+TF-TuM4, 5
Wolf, P.J.: TF-MoA10, 4
Won, C.: MI+TF-WeA9, **19**
Wong, M.S.: TF-WeP10, 13; TF-WeP26, **15**
Wu, Y.Z.: MI+TF-WeA9, 19
— X —
Xiao, Z.G.: PS+TF-TuM3, 5
— Y —
Yamada, I.: TF-WeM2, 10
Yamazaki, F.: TF-WeP31, 16
Yan, M.: TF-TuM3, 6
Yanagi, H.: TF-TuA3, 8
Yang, M.C.: TF-WeP10, 13; TF-WeP26, 15
Yang, T.S.: TF-WeP10, 13
Yaran, M.K.: TF-WeP30, 16
Yatabe, T.: TF-TuM7, 6
Yeadon, M.: MI+TF-WeA4, 18; TF-ThA7, **25**
Yi, S.S.: TF-WeP15, **14**; TF-WeP16, 14
Yokogawa, Y.: TF-WeP3, 12
Yoon, Y.J.: TF-WeP27, 16
Young, M.R.: TF2-WeA4, 21
Yu, J.: TF-ThA7, 25
Yu, Y.H.: TF-FrM10, 28
— Z —
Zabeida, O.: TF-WeM5, **10**
Zabinski, J.S.: TF+MM-WeA3, 20
Zambano, A.: MI+TF-WeA4, 18
Zangari, G.: MI+TF-WeA1, 18
Zapata-Torres, M.: TF-WeP7, 12
Zeipl, R.: TF-WeP20, 15
Zhang, Z.: MI+TF-WeA4, **18**
Zhao, H.W.: MI+TF-WeA9, 19
Zhou, X.W.: MI+TF-WeA10, **19**