The Science of Micro-Electro-Mechanical Systems Topical Conference

Room 610 - Session AS+MI+VM-MoM

Magnetic Recording: Chemical Integration and Tribology

Moderator: R.L. Opila, Bell Labs, Lucent Technologies

8:20am **AS+MI+VM-MoM1 Disk Drive Chemical Integration: Influence of Outgassing on Stiction**, ***D.E. Fowler***, *R.H. Geiss, E. Ghelichkhani,* Maxtor Corporation **INVITED**

Beyond optimizing the magnetic read/write sensor and the magnetic storage media, the successful introduction of a new disk drive product requires the integration of the best electronics and a great mechanical design to surround this magnetic interface. However, all of this effort will be for naught, if the disk drive cannot spin-up because the interface is stuck together or if contamination at the interface causes the sensor-to-media spacing to be a few nanometers greater than the designed fly height. Failure to spin-up can be the result of stiction and, in some cases, a phenomenon called fly stiction. We briefly discuss the distinguishing physical features of stiction induced by the disk lubricant as compared to stiction induced by in-drive outgassing, before focussing on outgassing-induced fly stiction. Various analytical methods have been used to identify the important outgassing sources and materials within the drive. The formation of liquid droplets on the read/write sensor during drive operation has been documented as an important contributor to increased stiction of the sensor-to-media interface following a period of nonoperation. We describe a real time visualization setup which monitors these processes in experimental, but fully functioning disk drives. This offers the opportunity to study the phenomenon and the mechanisms of fly stiction in a realistic drive environment. Results of these visualization experiments are presented. The goal of these studies is to develop a low-stiction interface through the optimization of the chemical integration of the drive. This allows the high-performance magnetic interface to function according to its design.

9:00am **AS+MI+VM-MoM3 The Evolution of the Corrosion Process on Thin-Film Media**, ***J. Ying***, *T. Anoikin, C. Martner,* MMC Technology Inc.

Thin-film hard disks have been exposed to elevated temperature/humidity, and dilute acidic vapor environment. These tests are designed to simulate possible galvanic corrosion, which, for the thin-film media, is characterized by the formation of Co and Ni containing corrosion nodules. The evolution of the corrosion process was elucidated by inducing different degrees of corrosion on the media, and these distinct corrosion stages were characterized morphologically by SEM and chemically by AES compositional analysis. In addition, an XPS chemical state study on the reactivity of Co, Cr, and Ni to ambient and chlorinated environments was conducted. A probable galvanic corrosion mechanism is proposed to understand the chemistry observed during the evolution of the corrosion process. In particular, the effects of ionic contaminants as corrosion accelerators and the role of the Cr underlayer as a corrosion-preventing barrier layer are discussed.

9:20am **AS+MI+VM-MoM4 Tribochemistry of Monodispersed ZDOL with Hydrogenated Carbon Overcoats**, ***C.-Y. Chen***, *W. Fong,* University of California, Berkeley*; D.B. Bogy,* University of California, Berkeley, U.S.

Tribo-chemical studies of the lubricant molecular weight effect on the tribology of the head/disk interface (HDI) were conducted using hydrogenated (CHx) carbon disks coated with ZDOL lubricant. The studies involved drag tests with uncoated and carbon-coated Al2O3-TiC sliders and thermal desorption experiments in an ultra-high vacuum (UHV) tribochamber. The studies showed that the lubricant interaction with the carbon overcoat varies as a function of lubricant molecular weight. The friction coefficient increases as the molecular weight increases. The higher friction is due to the higher viscosity. The friction and catalytic decomposition mechanisms of ZDOL are described. In general, the PFPE polymers are decomposed by chain scission involving the breakage of the backbone bonds to yield free-radical segments. Chain scission can occur by three mechanisms: (1) random degradation, (2) depolymerization, and (3) weak-link degradation. Our studies further support previous observations that catalytic reactions occurred at the endgroup functionals. The lower number of endgroup functionals for ZDOL with higher molecular weight reduces the possibility of the occurrence of catalytic reactions. Moreover, the ZDOL desorbed peak temperatures shifted to lower temperatures with increasing molecular weight in thermal desorption tests. The spreading diffusion coefficient of ZDOL decreases with increasing molecular weight. As the mobility of the lubricant chain decreases, the desorption energy needed to break the lubricants increases, resulting in higher desorption peak temperatures. In addition, the longer chain length of the higher molecular weight ZDOL causes higher degrees of crosslinking. The crosslinking restricts chain mobility and causes an increase in the desorption peak temperatures.

9:40am **AS+MI+VM-MoM5 Thermal Effects on Magnetic Head/Disk Interface Materials**, ***R. Koka***, Read-Rite Corp.*; L. Zhang,* Seagate Technology, Singapore **INVITED**

The materials interacting at the head/disk interface of a rigid disk drive are primarily the disk carbon overcoat, lubricant on the disk, and the head ceramic,Al2O3.TiC. The interface materials can be subjected to high, localized temperatures when the head is flying or sliding on the disk or when wear debris is trapped in the interface. The head or disk by itself can be exposed to high temperatures during the manufacturing process. This presentation addresses some of the changes that occur in the interface materials when they are individually subjected to high temperatures. Raman spectroscopy of thermally annealed disks will be presented to show that the carbon overcoat tends to become slightly graphitic. At 350C in air, the overcoat oxidizes rapidly and completely disappears. The widely used PFPE lubricants (ZDOL & AM) used on disks, degrade at high temperatures (~350C). In the presence of Lewis acids, the degradation process occurs at lower temperatures (~200C) and the rate of degradation is very high. The products of thermal degradation are different for the two lubricants because of the functional end groups. With respect to tribology, a head made of a passive ceramic such as SiC tends to perform relatively better than a material such as Al2O3.TiC, which is known to be an aggressive catalyst for lube degradation. Annealing of the Al2O3.TiC head ceramic shows that around 350C, carbon diffuses from the TiC grains and titanium oxides are formed. The diffused carbon is amorphous with a mixture of sp2 and sp3 bonds and it becomes nanocrystalline graphite above 600C. Thin, diamond-like, carbon coatings (60A thick) on the surface of the head effectively protect the Al2O3.TiC from oxidation and carbon diffusion at temperatures below 500C. A few examples of disk wear and smear formations on heads and disks will be presented. Some similarities between the Raman spectra of smears on heads and annealed disk overcoats and degraded lubricant will be discussed.

10:20am **AS+MI+VM-MoM7 The Process Induced Changes on the Co-alloy Films and the Tribological Effects on Magnetic Recording Heads**, ***Y.S. Chaug***, *R. Adams,* Storage Technology Corporation

The ferromagnetic alloys of Co-metal systems are soft magnetic materials having large saturation magnetization and low coercive force. Sputter deposited Co@sub 1-x@(Zr,Ta)@sub x@ (0.05<x<0.16) amorphous films have been used as magnetic pole material in magnetic inductive heads for its zero magnetostriction. In the wafer process, the Co@sub 1-x@(Zr,Ta)@sub x@(CZT) films were patterned through the photolithography process and then treated with an oxygen plasma for cleaning. The surface changes on the processed CZT surface were studied using x-ray photoelectron spectroscopy. The migration of Co ions to the CZT surface was found after the oxygen plasma treatment. Atomic force microscope, scanning electron microscope and Nano-Triboscope were used to examine the changes of the Co rich CZT surface in a high humid environment. The process induced changes on the CZT surface which impacted the ABS (air bearing surface) lapping process in manufacturing the magnetic inductive heads. The tribology of the magnetic recording heads using CZT as magnetic pole will be discussed.

10:40am **AS+MI+VM-MoM8 Study of Tribochemical Processes at the Head-disk Interface Using Photoemission Electron Microscopy**, ***S. Anders***, *A. Scholl, F. Nolting,* Lawrence Berkeley National Laboratory*; W. Fong, C.-Y. Chen,* University of California, Berkeley*; D.B. Bogy,* University of California, Berkeley, U.S.*; C.S. Bhatia,* SSD/IBM*; J. Stohr,* IBM Almaden Research Center

Photoemission electron microscopy (PEEM) has been applied to study the tribochemical processes at the head-disk interface of magnetic storage devices. High resolution PEEM imaging is based on several contrast mechanisms (topographical, elemental, chemical, and various forms of polarization contrast) which makes it a unique tool for the study of tribochemical processes. We have studied surfaces of hard disks and sliders after various kinds of wear tests performed in ambient atmosphere and UHV. It was observed that the disk surface in the wear tracks is chemically modified if visible wear occurred and if a lubricant was present. In the case of unlubricated disks no chemical modifications were observed but a reduction in the hard carbon overcoat thickness. The chemical modifications consist of lubricant oxidation and fluorine removal. The

lubricant oxidation and fluorine removal is enhanced with enhanced wear. It was found that degraded lubricant is transferred to the sliders and accumulated in scratches of the slider surfaces. The hard carbon overcoat on sliders was found to be reduced in thickness after the wear tests, but not chemically altered.

11:00am **AS+MI+VM-MoM9 Tribo-Chemistry of the Head-Disk Interface in Hard Disk Drives**, ***D.B. Bogy***, University of California, Berkeley, U.S.*; C.S. Bhatia,* IBM SSD*; C.-Y. Chen, W. Fong,* University of California, Berkeley **INVITED**

Tribo-chemical studies of the lubricant thickness effect on the tribology of the head/disk interface (HDI) were conducted using hydrogenated (CHx) carbon disk samples coated with perfluoropolyether ZDOL lubricant. The studies involved drag tests with uncoated and carbon-coated Al@sub 2@O@sub 3@-TiC sliders and thermal desorption experiments in an ultra-high vacuum (UHV) tribochamber. The studies showed that the lubricant interaction with the carbon overcoat varies as a function of lubricant thickness. Wear durability improves considerably for thicknesses greater than a monolayer. However, in the sub-monolayer thickness regime, the adhesion of the lubricant to the carbon overcoat is much stronger, as indicated by the fact that a much higher temperature is required to desorb the lubricant. When the lubricant thickness is around or above a monolayer, cohesion among the lubricant molecules plays a greater role and a much lower temperature is needed for lubricant desorption. In addition, we observed that hydrogen evolution from CHx overcoat initiates lubricant catalytic decomposition, forming CF3 and C2F5. The generation of HF during the thermal desorption experiments provides the formation mechanism of HF, which is the necessary component for catalytic reaction.

11:40am **AS+MI+VM-MoM11 Phase Transitions in Two-dimensional Ferroelectric Films**, ***C.N. Borca***, *J. Choi, S. Adenwalla, P.A. Dowben, M. Poulsen,* University of Nebraska, Lincoln*; J.L. Robertson,* Oak Ridge National Laboratory*; V.M. Fridkin, S.P. Palto, N. Petukhova, S.G. Yudin,* Russian Academy of Science*; S. Ducharme,* University of Nebraska, Lincoln

We studied ferroelctric copolymer films of vinylidene fluor ide with trifluoroethylene, P(VDF-TrFE) 70:30. The films exibit ferroelectric switching properties and can be used in a variety of piezoelectric devices. In addition to the first order ferroelectric to paraelectric bulk transition at 80 degrees C, we report two other phase transitions. One appears at 20 degrees C and is related entirely to a surface ferroelectric transition.@footnote 1@ The third transition around 150 degrees K is due to a stiffening of the lattice and a change in the bulk electronic structure. For P(VDF-TrFE), there is a negligible density of states at the Fermi level making this phonon related transition very unusual. This last transition was observed using neutron diffraction, @footnote 2@ X-ray diffraction, photoemission spectroscopy and EELS. The effective Debye temperature decreases from a value of about 250 K to 50 K with increasing temperature across the 150 K lattice stiffening transition. @FootnoteText@ @footnote 1@J. Choi, P.A. Dowben, S. Pebley, A.V. Bune, S. Ducharme, V.M. Fridkin, S.P. Palto, N. Petukhova, Phys. Rev. Lett. 80, 1328 (1998) @footnote 2@C.N. Borca, J. Choi, S. Adenwalla, Stephen Ducharme, P.A. Dowben, Lee Robertson, V.M. Fridkin, S.P. Palto, and N. Petukhova, Appl. Phys. Lett. 74, 347 (1999).

Thin Films Division

Room 620 - Session TF+VM-MoM

Advances in Hard and Superhard Coatings I

Moderator: F. Sequeda, Universidad del Valle, Columbia

8:20am **TF+VM-MoM1 Advances in Hard and Superhard Coatings for Tribological Applications**, ***A. Matthews***, *A. Leyland,* University of Hull, UK **INVITED**

Over the past twenty years there has been considerable progress both in the development of advanced coating processes and in the scientific understanding of tribological mechanisms. Typically these developments have occurred completely separately, to the extent that coatings researchers may seek to develop a new coating with a specific extreme property (such as high hardness) driven primarily by scientific curiosity, rather than a desire to fulfil an identified tribological need. Usually that need will (for example) require an enhanced range of properties (such as hardness, toughness and resistance to environmental degradation). Coatings researchers are now increasingly recognising this need and are fulfilling it through several exciting developments. These include multi-layered and nanocomposite coatings which combine high hardness (H) with a relatively low elastic modulus (E) (to provide increased toughness). These coatings thus minimise the E/H ratio - which is known to be a parameter which is closely related to wear resistance. Other developments involve so-called 'hybrid' or 'duplex' combinations of processes to fulfil specific tribological needs. We are now in sight of achieving functionally-graded coating structures which combine tough and stress-equalised bulk properties, together with the extreme hardness, thermal and chemical properties of the exterior. The paper discusses the developments which have been necessary to achieve this - such as optimisation of compound compositions in the case of nanostructured composites based on nitride, carbide and boride phases. Also in the case of hard oxide ceramics, mention is made of growth modelling studies and plasma process developments to achieve the desired phases.

9:00am **TF+VM-MoM3 Ion-Assisted Filtered Cathodic Arc Deposition (IFCAD) Technology for Production of Superhard Thin-Film Coatings**, ***M.L. Fulton***, Ion Arc Corporation

A new Ion-Assisted Filtered Cathodic Arc Deposition (IFCAD) system has been developed for low temperature production of superhard thin-film coatings. Only ions within a well defined energy range arrive at the substrate surface depositing thin-films with excellent mechanical and optical properties.@footnote 1@ The new IFCAD system consists of a cylindrical rotary deposition chamber with two (or four) Filtered Cathodic Arc (FCA) sources, each associated with an end-Hall Ion-Assisted-Deposition (IAD) ion gun.@footnote 2@ By coupling IAD with FCA the development of cost effective deposition processes for applying superhard advanced thin-film materials such as: Amorphous Diamond-Like-Carbon (A-DLC); Aluminum Oxide (Al2O3); Aluminum Nitride (AlN); Carbon Nitride (C3N3); Titanium Nitrite (TiN); Titanium Nitride Carbide (TiCN); Titanium Oxide (TiO2: Rutile); and others in multi-layer thin-film structures suitable for tribological and electro-optical applications is now feasible. The IFCAD film properties are superior to other processes at elevated deposition temperatures, for example: the A-DLC thin-films have a micro-hardness in excess of 50 GPa (Diamond = 100 GPa); and the amorphous Al2O3 films have a hardness in excess of 20 GPa (bulk sapphire is 35 GPa). This new IFCAD technology has been included in advanced commercial, military and space development programs, such as: EUV mirrors; plastic and glass lens coatings for optical systems; wear resistant coatings on various metal substrates; and ultra smooth, durable, surface coatings for injection molds. @FootnoteText@ @footnote 1@ P. J. Martin, R. P. Netterfield, A. Bendavid, and T. J. Kinder, "The deposition of thin films by filtered arc evaporation," Surface and Coatings Technology, 54/55 (1992) 136-142. @footnote 2@ M. L. Fulton, "Application of ion-assisted-deposition using a gridless end-Hall ion source for volume manufacturing of thin-film optical filters," in Optical Interference Coatings, Florin Abeles, Editor, Proc. SPIE 2253, (1994) 374-393.

9:20am **TF+VM-MoM4 High Rate Reactive DC Magnetron Sputtering of Al Oxide and W Oxide Thin Films; Large Area Coatings**, ***M.K. Olsson***, Fraunhofer Institute for Solar Energy Systems, Germany*; K. Macák,* Linköping University, Sweden

Recently we reported stable high-rate deposition of Al oxide with any composition, including stoichiometry, utilizing a conventional reactive DC magnetron sputter system in laboratory scale.@footnote 1,2@ Due to the proper system geometry, including large enough target-to-substrate distance, and/or sufficient process gas pressure, it was possible to keep the target in the metallic mode by taking advantage of the scattering of the sputtered Al atoms through the inert gas. Moreover, the relatively high working pressure caused an increase in the back-deposited Al atoms to the non-eroded areas of the target, thus keeping these areas conducting, thereby avoiding arcing, without requiring the use of any other devices. We have applied this concept to a sputtering system considered for large area thin film production. A construction for increasing the cathode-to-sample length was designed with our technical possibilities in mind. After installation the desired O/Al arrival ratio was obtained and it was possible to produce stoichiometric aluminum oxide with an order-of-magnitude higher deposition rate. Choice of a sufficient pressure was crucial for the long-term stability of the process.@footnote 3@ Once the target is in the metallic state one may increase the growth rate linearly by increasing the target current. However, the experiments and analysis of the deposition process based on MC simulation of sputtered particles transport extended by Rossnagel's model of gas heating@footnote 4@ confirmed that the efficiency of the gas scattering process is for heavy elements partially eliminated at high discharge currents. To deal with this, we applied our concept of proper choice of process conditions for sputtering to less reactive materials with relatively high atom mass. It was possible to improve the sputtering condition for making films of stoichiometric

amorphous W oxide at relatively high target current.@footnote 5@ @FootnoteText@ @footnote 1@M. Kharrazi Olsson, K. Macák, U. Helmersson, and B. Hjörvarsson, J. Vac. Sci. Technol. 16, 639 (1998). @footnote 2@K. Macák, T. Nyberg, P. Macák, M. Kharrazi Olsson, U. Helmersson, and S. Berg, J. Vac. Sci. Technol. 16, 1 (1998). @footnote 3@M. Kharrazi Olsson, K. Macák, W. Graf, Submitted. @footnote 4@S.M. Rossnagel, J. Vac. Sci. Technol. 6, 19 (1988). @footnote 5@M. Kharrazi Olsson, K. Macák, Submitted.

9:40am **TF+VM-MoM5 Elastic and Plastic Behaviors of Al/TiN Multilayered Thin Films Evaluated by Nanoindentation**, ***E. Kusano***, *Y. Sawahira, N. Kikuchi, H. Nanto, A. Kinbara,* Kanazawa Institute of Technology, Japan

Elastic and plastic behaviors of multilayer films of Al (Young's modulus:70GPa) and TiN(Young's modulus:350-400GPa) have been investigated for various layer numbers with different total Al thicknesses in order to reveal the role of hard TiN and soft Al layers on nanomechanical properties in multilayered films. Both Al and TiN layers were deposited by dc magnetron sputtering. Aluminosilicate glass was used as substrate. The number of layers prepared was 4, 20, and 40 for the total Al thickness of 100-500nm. The total thickness of TiN layers was kept at 500nm, including the top layer of 250nm. The microhardness and Young's modulus of the films were evaluated by nanoindentation. The energies consumed for elastic and plastic deformations were calculated from the load-displacement curve obtained by the indentation. The microhardness decreased with increasing the total Al thickness. The hardness enhancement by the multilayer structure was observed for 20- and 40-layer films with Al layer thicknesses of 10 or 5nm (the total thickness of 100nm) and TiN layer thicknesses of 26 and 13nm. The energy dissipated during the indentation also increased with the total Al thickness. The dissipated energy for films with 40 layers of Al/TiN was smaller than that for the films with 4 or 20 layers of Al/TiN for all Al thicknesses. In contrary, the elastic energy was independent both of the total Al thickness and of layer numbers. As a result, the ratio of dissipated energy to the loaded energy during the load/unload of the indentation yielded a minimum at an Al thickness of 100nm for 40-layer films. The ratio at the minimum was about 20% smaller than that of the monolithic TiN film. This means that the 40-layer film with a total Al thickness of 100nm is more elastic than the monolithic TiN. It is concluded that the enhancement in the film microhardness for multilayered films with thin Al layers is related to the decrease in the dissipated energy.

10:00am **TF+VM-MoM6 Gas-phase Chemistry in Up-scaled Plasma Enhanced MOCVD of TiN and Ti(C,N) on Plasma Treated Tool Steel**, ***J.P.A.M Driessen***, *A.D. Kuypers,* TNO Institute of Applied Physics, The Netherlands*; J. Schoonman,* Delft University of Technology, The Netherlands

In this paper, the deposition of TiN and Ti(C,N) in a relatively large scale reactor vessel is discussed. Tetrakis(dimethylamine)titanium (TDMAT) and tetrakis(diethylamine) titanium (TDEAT) were used for the purpose of depositing TiN and Ti(C,N) at low temperatures. In large scale systems, homogeneous reactions dominate the deposition process resulting in non-uniform and non-adherent coatings. However, in this study, favourable gas-phase conditions for deposition of Ti(C,N) from in a pulsed DC-plasma have been determined, making use of mass and optical spectroscopy. Decomposition of TDMAT in a pure hydrogen plasma results in the favourable cleavage of dimethylamine from TDMAT but prevents the formation of Ti(C,N) due to the lack of nitrogen and carbon. Addition of N@sub 2@ to the hydrogen plasma results in the formation of NH@sub x@ (1@<=@x@<=@4), opening transamination pathways. Results suggest that transamination plays an important role in the gas-phase of our system. Furthermore, these results were compared with those obtained from using ammonia. However, the depletion of TDMAT by interaction with nitrogen in a H@sub 2@(85%) - N@sub 2@(15%) plasma proceeds in a mechanistic step with a rate constant of k = 4.7 x 10@super -14@ cm@super 3@ mol@super -1@sec@super -1@. Nevertheless, seemingly high quality Ti(C,N) coatings were deposited on blank WN1.2370 tool steel and WN 1.2379 treated in a N@sub 2@/H@sub 2@ plasma. XRD analysis of the plasma treated substrate indicates the presence of CrN, among other species in the top surface layer. These multi-layer coatings, deposited at temperatures between 473 K and 698 K, increased in surface roughness, however, showed good adherence and optimum hardness. Hardness values varied from 1600 Hv to 2000 Hv.

10:20am **TF+VM-MoM7 Effect of Ion-to-neutral Ratio and Ion Energy on Structure and Properties of Boron Nitride Thin Films**, ***M.U. Guruz***, *Y.W. Chung, V.P. Dravid,* Northwestern University

Boron nitride thin films were deposited by dc reactive magnetron sputtering using a B@sub 4@C target in a single cathode chamber. The films were grown on Si (001) wafers, held at ambient temperature. The energy of the ions arriving at the substrate surface was determined by the applied bias. Additionally, an external coil assembly was placed outside the chamber, allowing modification of the magnetic field around the substrate. By varying the field strength, the ion flux on the substrate was enhanced. Thus, the ion-to-neutral ratio and the ion energy were independently controlled during deposition. The effects of these two parameters on the resulting film microstructure were investigated by transmission electron microscopy (TEM), atomic force microscopy (AFM), Fourier transform infrared spectroscopy (FTIR) and nanoindentation. These results and the effects on the formation of cubic boron nitride will be presented.

10:40am **TF+VM-MoM8 Mechanical Properties of Cubic Boron Nitride Thin Films Synthesized by ECR PECVD: Influence of Deposition Conditions**, ***M.P. Delplancke-Ogletree***, *M. Ye,* Université Libre de Bruxelles, Belgium

Cubic boron nitride containing thin films were deposited on (100) Si and steel substrates by electron cyclotron resonance plasma enhanced chemical vapor deposition. The films contain at least 55% of the cubic phase and are 0.5 µm thick. We investigated the dependence of hardness, stress, adherence, friction coefficient and wear resistance as a function of deposition parameters. The studied parameters are substrate bias, gas mixture composition, substrate temperature and processing pressure. These parameters are also correlated to the plasma characteristics measured by Langmuir probe, and mass spectrometer. Nanoindentation, scratch test, cantilever deflection, and ball-on-disk methods were used to evaluate quantitatively the mechanical properties. Films containing 55% of cubic phase are adherent to the two types of substrates, and have stress below 5 GPa.

11:00am **TF+VM-MoM9 Plasma Assisted Physical Vapour Deposition of BN by DC Pulsed Sputtering of a B@sub 4@C Target**, ***L.A. Gea***, *G. Ceccone, F. Rossi,* European Commission Joint Research Centre, Italy

Boron nitride coatings were deposited on Si (100) polished crystals by DC pulsed magnetron sputtering of a B4C target with auxiliary microwave Distributed Electron Cyclotron Resonance (DECR) plasma.The substrates were biased by an independent R.F. source. Characterization of the plasma was undertaken as a function of the total pressure, the nitrogen gas content and the microwave power. The presence of the various species was identified with Optical Emission Spectroscopy.Mass Spectrometry was used to determine the ion energy distribution while the plasma potential and the plasma densities were measured with a single Langmuir probe.The coatings have been characterized by Scanning Electron Microscopy (SEM), Auger Electron Spectroscopy (AES), and Fourier Transformed Infrared Spectrometry (FTIR)

11:20am **TF+VM-MoM10 Field Emission from Flat, Diamond-like Carbon Films Characterized by Scanning Force Microscopy**, *T. Inoue,* Electrotechnical Laboratory, Japan*;* ***D.F. Ogletree***, *M. Salmeron,* Lawrence Berkeley National Laboratory

Thin films of various diamond-like and carbon based materials on flat cathodes show significant field emission at relatively low voltages, but the exact emission mechanisms are not well understood. Non contact scanning force microscopy with a conductive tip in vacuum, used in the scanning polarization force mode (SPFM) , can detect and characterize emission sites with 100 nm lateral resolution. The SFM tip serves as an anode. It can be scanned over the surface to simultaneously measure local emission currents and local work functions. The tip-sample spacing and the tip bias can be varied to investigate mechanisms. Contact SFM images of emitting regions show local topography, reveal the presence of asperities, and characterize sample conductivity. For one type of CVD cathode material on an Si substrate, the emission sites were found to be (a) ~ 1 micron in diameter (b) not associated with asperities at the cathode-vacuum interface (c) not associated with low work function regions, and (d) semiconducting with a large band-gap. I-Z data indicate that emission takes place within the film, or at the film-substrate interface, rather that at the film-vacuum interface. Single-site emission currents were strongly modulated on a ~ 1 ms time scale, possibly due to charge trapping. Surface potential shifts were correlated with the emission current fluctuations.

11:40am **TF+VM-MoM11 Effect of Nondiamond Carbon on the Electron Transport Path of Field-emitted Electrons from Undoped Polycrystalline Diamond Films**, ***J.Y. Shim,*** Yonsei University, Republic of Korea*; K.M. Song,* Konkuk University, Republic of Korea*; H.K. Baik,* Yonsei University, Republic of Korea, KOREA

Diamond has attracted considerable interest recently as a promising field emitter material because of its important properties such as NEA, high thermal conductivity, and low field electron emission. Among the properties, understanding the origin of low field emission is a key factor for the application of diamond to a field emitter. Many investigations have been extensively drawn to clarify the origin of low field emission behavior of diamond. Suggested models explaining the low field emission behaviors up to now are the discontinuous graphitic inclusions in CVD diamond matrix, the defect induced subbands, and the surface emission from metal-diamond-vacuum interface. Besides, there have been several reports on the importance of the electron transport through the substrate/diamond interface. However, the origin of the low field emission behavior is still unclear, and those may be different between diamond crystal and CVD diamond films. It is expected that the field emission mechanism of undoped polycrystalline diamond films can be changed by the structural properties. The present study is mainly focused on the electron transport through the substrate-diamond interface and the diamond layer, and the resulting field emission mechanism of undoped polycrystalline diamond films with different structural properties. In order to examine field emission properties of undoped polycrystalline diamond films, we have prepared the diamond films with different structural properties and/or different substrate-diamond interfaces. It was observed that transport paths of field-emitted electrons could be clearly identified from the spatial distributions of emission sites and the nondiamond carbon content obtained from the diamond films, and the characteristics of the substrate-diamond could modify the field emission properties. From the present study, a possible field emission mechanism for the undoped polycrystalline diamond films is suggested.

Magnetic Interfaces and Nanostructures Technical Group
Room 618/619 - Session MI+VM+AS-MoA

Magnetic Recording: Media and Heads

Moderator: D.E. Fowler, Maxtor Corporation

2:00pm **MI+VM+AS-MoA1 Spectro-Microscopy of Magnetic Materials Using Polarized Soft X-Rays**, ***J. Stohr***, IBM Almaden Research Center **INVITED**

The talk discusses the motivation for and challenges of obtaining magnetic information for ferromagnetic and antiferromagnetic systems on a length scale below 100nm. It reviews the principles of linear and circular x-ray magnetic dichroism spectro-microscopy and presents state-of-the-art results (20nm resolution) obtained with a dedicated soft x-ray photoelectron emission microscope (PEEM) installed on the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory. Results include studies of the antiferromagnetic domain structure at the surface of LaFeO3(100), NiO(100) and polycrystalline NiO and the ferromagnetic domain structure in hard/soft magnetic tunnel junctions. Future plans to reach a spatial resolution below 10nm will also be discussed.

2:40pm **MI+VM+AS-MoA3 Micromagnetic Properties and Recording Performance in High Density Magnetic Recording Media**, ***T. Suzuki***, *G. Lauhoff,* Toyota Technological Institute, Japan **INVITED**

In order to increase areal recording density, much effort has been made to improve magnetic charactreristics and microstructure in magnetic recording media. In order for decreasing noise and for realizing a sharp transition between written bits, an inter-granular exchange coupling must be lowered, which enhances a thermal instability of magnetization. Given this conditions, it is vitally important to understand the role of magnetic activation volume or magnetic coupled region in conjunction with micromagnetics, which plays a key role in noise mechanisms. The present study is to discuss activation volume in longitudinal magnetic recording media of various types including CoCrPtTa thin-films and granular-type recording media. The activation volume is evaluated based on the time decay of magnetization at a certain field. A novel method to define the activation volume is proposed. The Barkhausen volume is estimated through the field-sweep-rate dependence of coercivity. It is found that the activation volume, which is of the order of 10@super -18@ cm@super -3@, decreases with applied field, then levels off, and starts increases at fields beyond coercivity. This trend is found for all the high density recording media of CoCrPtTa and granular-type media under consideration. The recording noise is found to be closely related to activation volume. Measurments of @delta@M curves suggest that lesser the granular-exchange-coupling is, the smaller the activation volume becomes.

3:20pm **MI+VM+AS-MoA5 Sub 50 nm Planar Magnetic Nanostructures Fabricated by Ion Irradiation**, *T. Devolder,* ***C. Chappert***, IEF/Université Paris Sud, France*; Y. Chen,* L2M Bagneux/CNRS, France*; H. Bernas,* CSNSM/Université Paris Sud, France*; J.-P. Jamet, J. Ferré,* LPS/Université Paris Sud, France*; E. Cambril,* L2M Bagneux/CNRS, France **INVITED**

Areal density enhancement is a major challenge in magnetic recording. Near field magneto-optical techniques are one fast-developing attempt to respond. At bit density values above 65 Gbits/in@super 2@, a most drastic requirement will be to write stable bits with nanometer wall jaggedness, at very precise locations on the disk. Patterned media could be a promising response to this problem.@footnote 1@ However, surface roughness, and polarization dependent effects due to abrupt changes in optical index, will likely deteriorate the signal to noise ratio. Through interface mixing, light ion (He+) irradiation can modify in a precisely controlled way the magnetic properties of multilayers, with negligible change of surface roughness and optical indices. In (Co/Pt) multilayers with perpendicular easy magnetization axis, the anisotropy decreases with irradiation, which first reduces the coercive force, then induces in-plane magnetization. Patterning only the magnetic properties can then be obtained by irradiation through a lithographic PMMA resist mask.@footnote 2@ Using SiO@sub 2@ masks, we have fabricated regular arrays of such irradiation-patterned nanostructures with sizes down to 30 nm. Different configurations such as hard (resp. soft) nanostructures in soft (resp. hard) media have been obtained and characterized using far field magneto-optical microscopy. Special attention has been devoted to the study of the transition zone between irradiated and protected areas, and its effect on magnetization reversal. The technique may be a powerful tool for ultrahigh density magnetic recording applications. @FootnoteText@ @footnote 1@ S. Chou et al., Data Storage 35 (1995). @footnote 2@ C.Chappert et al., Science 280, 1919 (1998).

4:00pm **MI+VM+AS-MoA7 Crystallographic Texture and Stress in Co-Based Magnetic Recording Media and Underlayers**, ***B.M. Clemens***, *G. Khanna,* Stanford University **INVITED**

The magnetic and recording properties of Co-based media are a strong function of crystallographic texture and stress in the media layers, which are in turn strongly influenced by the texture and stress of the Cr underlayer. The anisotropy in strain and the distribution of c-axes in the hcp Co alloy media can determine the magnetic hysteresis anisotropy through magnetostrictive and magnetocrystalline effects. The processing conditions and mechanical texture grooves in the NiP/Al substrate strongly influence the microstructural and mechanical properties of the Cr and Co films. We report x-ray studies of the texture and strain in Cr and Co-based films sputter deposited over a range of temperatures and substrate biases on smooth and mechanically textured substrates. All films showed an in-plane compressive stress. The magnitude of the stress in Cr and Co films grown without a substrate bias decreases with decreasing growth temperature, consistent with thermal mismatch stress. The stress in the Cr films grown with a -300V bias was greater than that of unbiased films, and independent of growth temperature. This suggests that the bias-induced stress is close to the yield stress of the film. However, the biased Co films show a decrease in compressive stress with decreasing temperature. For mechanically textured substrates, the stress measured in the direction perpendicular to the texture grooves was less compressive relative to the parallel direction in both Cr and Co. However, the stress anisotropy vanishes in smooth substrates. A simple strain relaxation model is used to explain the observed Cr stress values in the textured disks. The anisotropic strain in the Cr may also account for the observed preferential alignment of Co c-axes along the grooves. Calculation of the magnetoelastic and magnetocrystalline energies predicts that Co stress anisotropy and the preferential alignment of Co c-axes along the grooves both contribute to the observed hysteresis anisotropy.

4:40pm **MI+VM+AS-MoA9 Noise in GMR Recording Heads**, ***H.T. Hardner***, *M.B. Hurben,* Seagate Technology **INVITED**

Magnetoresistive sensors exploit a close coupling between magnetization and resistance to convert changes in magnetic field to an electrical signal. Thus, the enhanced sensitivity of the giant magnetoresistive (GMR) materials to magnetic field is accompanied by larger electrical noise due to magnetization noise. A magnetic contribution to 1/f noise originates in thermal fluctuations in magnetization. This is a concern for sensors intended for low frequency applications rather than for magnetic recording heads due to the very high frequencies at which the heads operate. However, discrete steps in the resistance can also appear due to large discrete changes in domain structure. When these steps occur as a continuous magnetic signal is applied to the device they are called Barkhausen noise. In small enough devices discrete magnetoresistive steps can be observed even at fixed magnetic field. While the sensitivity of the resistance to changes in magnetization is proportional to the magnetoresistance, the propensity for complex domain structure with fluctuations between multiple metastable states varies by material, processing, and design. The suppression of these fluctuations to create a device with a single stable magnetization state is a key goal in the design and manufacture of magnetoresistive recording heads. This talk will provide some brief background on how recording heads are stabilized as well as an overview of resistance noise data from GMR devices including comparisons of different materials and the use of resistance noise measurements to study domain structure. Recent results on resistance noise in spin-valve recording heads both at the finished stage and during wafer processing will be presented along with discussion of how a detailed study of electrical noise can help identify stability problems. Analysis of both time and frequency domain data will be considered.

Thin Films Division

Room 620 - Session TF+VM-MoA

Advances in Hard and Superhard Coatings II

Moderator: B. Holloway, College of William & Mary

2:00pm **TF+VM-MoA1 Characterization of PVD TiN/CN@subx@ and TiN/Si@sub3@N@sub4@ Multilayer Coatings**, ***Y.H. Chen***, *Y.W. Chung,* Northwestern University

TiN coatings are commonly used in various tribological applications for their wear resistance and inertness to steels. However, TiN coatings predominantly grow with a columnar grain structure. The columnar grain boundaries become the usual sites for crack initiation, resulting in earlier failure of TiN coatings (especially thick coatings). In our research, TiN/a-CN@subx@ and TiN/a-Si@sub3@N@sub4@ nanolayered superlattice coatings are developed to suppress the columnar structure. We used a-CN@subx@ and a-Si@sub3@N@sub4@ primarily to periodically interrupt and renucleate the growth of TiN. In addition, the amorphous layers may serve to suppress the transmission of dislocations from one TiN layer to another, thereby enhancing the hardness of the coating. Both coatings have been demonstrated to achieve hardness in the 50 GPa range, consistent with recent reports for high hardness of TiN/a-Si@sub3@N@sub4@ nanocomposites. The correlation between microstructure and mechanical properties of these coatings will be presented.

2:20pm **TF+VM-MoA2 Effects of Interface Mixing on Adhesion of Amorphous Carbon Films Synthesized by Variable-Energy Direct Carbon Ion Beam Deposition**, ***M.H. Sohn***, *S. Kim,* SKION Corporation

Using a variable-energy direct carbon ion beam deposition technique, thin amorphous carbon films were grown on silicon substrate. Interface modification was performed using C@super -@ energies in the range of 300-500 eV prior to the growth of the film to enhance adhesion of the film. By lowering the energy of the C@super -@ beam to 150 eV, amorphous carbon film was continuously grown after the interface modification. High-resolution electron microscopy illustrated that the silicon surface was severely damaged by 500 eV C@super -@ beam and the thickness of damage layer was about 15 nm. Carbon composition profile in silicon investigated by electron energy loss spectroscopy showed that 500 eV C@super -@ beam implanted carbon into silicon up to 30 nm in depth and carbon was mixed with silicon at this implanted region. Silicon L-edge study at the C/Si mixed region found C-Si bonding formation only at the surface of silicon over 2-3 nm-thick layers. The damage layer or C/Si mixing was not observed at 300 eV C@super -@ beam modification. Wear testing found that strong adhesion occurred in samples modified at 500 eV, which indicated complete mixing at the interface. At 300 eV, modified samples exhibited delamination failure, which indicated inferior adhesion of the films.

2:40pm **TF+VM-MoA3 Synthesis of Diamondlike Carbon Films with Superlow Friction and Wear Properties**, ***A. Erdemir***, *O.L. Eryilmaz, G. Fenske,* Argonne National Laboratory

In this study, we introduce a new diamond-like carbon (DLC) film providing friction coefficients of 0.001 and wear rates of 10-9 to 10-10 mm3/N.m in inert gas environments (e.g., dry nitrogen and argon). The film was grown on steel and sapphire substrates in a plasma enhanced chemical vapor deposition system using a hydrogen-rich plasma. Employing a combination of transmission electron microscopy, electron diffraction, Raman spectroscopy, and electron energy loss spectroscopy, we explored the structural chemistry of the resultant DLC films and correlated these findings with their friction and wear mechanisms. The results of tribological tests under a 10 N load (creating an initial peak Hertz pressure of 1 GPa on steel test pairs) and at 0.2 to 0.5 m/s sliding velocities indicated that a close correlation exists between the friction and wear coefficients of DLC films and the source gas chemistry. Specifically, films grown in source gases with higher hydrogen-to-carbon ratios had the lowest friction coefficients and the highest wear resistance. The lowest friction coefficient (i.e., 0.001 on a sapphire substrate) was achieved with a film derived from a gas mixture consisting of 25% methane and 75% hydrogen. The wear-debris particles found in and around the wear scars and tracks were analyzed by Raman spectroscopy and FTIR to elucidate the wear mechanism of DLC films. @FootnoteText@ *Work supported by the U.S. Department of Energy under contract W-31-109-Eng-38.

3:00pm **TF+VM-MoA4 Optical Characteristics of Carbon Nitride: Relationship with Mechanical Behavior and Possible Fullerene-like Microstructure**, ***V. Hajek***, *D. Poitras, D. Dalacu,* Ecole Polytechnique, Canada*; A. Bergeron,* Optical Coating Laboratory Inc.*; L. Martinu,* Ecole Polytechnique, Canada*; K. Rusnak, J. Vlcek,* University of West Bohemia, Czech Republic

Crystalline @beta@-C@sub 3@N@sub 4@ was predicted to exhibit extreme properties, such as hardness, comparable to that of diamond. Although the synthesis of the crystalline metastable phase has not been fully confirmed yet, already prepared "amorphous" CNx films possess very attractive characteristics. In our earlier studies we have shown that these films possess high hardness (up to 30 GPa), high elastic recovery (up to 85 %), and interesting tribological behavior. Such films prepared at temperatures above 200 °C, using magnetron sputtering, are predicted in recent literature to possess a fullerene-like microstructure. In our recent work we suggested to extend this model to account for hydrogen incorporation: Excessive amount (> 1 at. %) of hydrogen in the films is believed to inhibit crosslinking between graphite-like planes containing carbon and nitrogen, and thus to hamper formation of the fullerene-like microstructure. In the present work we focus on the optical properties of CN@sub x@ films studied by spectroscopic ellipsometry and spectrophotometry. Different dispersion relations such as Sellmeier, Cauchy and Drude-Lorenz oscillator were used to determine optical constants n and k. The optical behavior is related to the film microstructure and the film fabrication conditions. CN@sub x@ layers were deposited on Si substrates by reactive DC magnetron sputtering of graphite target in nitrogen plasma at a substrate temperature of 600 °C and at a substrate bias ranging from -300 to -700 V. Films were found substoichiometric in nitrogen (from 12 to 24 at. %), and a concentration of hydrogen between 1 and 5 at. % was revealed by elastic recoil detection (ERD) analysis. Increased hydrogen content was accompanied by lower hardness, elastic recovery and adhesion, by higher electrical resistivity (from 20 to 970 @ohm@cm), by formation of C-H and N-H bonds (FTIR), and by higher optical transparency in the VIS and IR regions. The latter characteristics are related to the variation of optical bandgap which increases with hydrogen incorporation.

3:20pm **TF+VM-MoA5 Preparation and Properties Enhancement of Silicon Carbonitride Films Using Reactive Magnetron Sputtering**, *X.-M. He, K.C. Walter,* ***M. Nastasi***, Los Alamos National Laboratory

Silicon carbonitride (Si(C,N)) films were synthesized on Si (100) and metal substrates by reactive d.c. magnetron sputtering with Ar as the sputtering gas and N@sub 2@ as the reactive gas. The composition and properties of the films were studied with respect to the influences of the bias voltage applied to substrates, the deposition temperature, and the gas flow ratio of N@sub 2@ to Ar (or F@sub N2@/F@sub Ar@). The Si(C,N) mechanical properties, hardness, fracture, and tribological properties, were observed to be highly depended on the processing conditions such as substrate temperature, the arrival ratios of ion to deposition atom, J@sub i@/J@sub a@, the negative bias voltage and F@sub N2@/F@sub Ar@. Under optimum conditions amorphous coatings with high wear resistance and harnesses as high as 40 GPa were prepared. The role of synthesis parameters on the structure, compositions, and mechanical properties will be discussed in detail.

4:00pm **TF+VM-MoA7 Bonding Structure and Optical Properties of Si-doped Diamond-like Films Synthesized by Plasma Immersion Ion Processing**, ***X.-M. He***, *K.C. Walter, M. Nastasi,* Los Alamos National Laboratory

Silicon-doped diamond-like carbon (DLC) films were prepared on Si(100), glass, and PMMA (polymethyl methacrylate) substrates at room temperature by using C@sub 2@H@sub 2@-SiH@sub 4@-Ar plasma immersion ion processing (PIIP) and their compositions were modified by changing deposition parameters of the gas composition and the negative bias voltages applied on the substrates. The influence of the Si dopant on the bonding structure and the properties of the DLC films were investigated by using ion beam analysis techniques, Raman shift, infrared spectroscopy, and by analyzing the measured density and hardness. The electrical and optical properties of Si-doped DLC films have been evaluated by the study of the electrical resistivity, the refractive index, the absorption coefficient, and the optical gap energy for the films. It was found that the variation of Si dopant was highly correlated with the changes of chemical bonding structure and properties. The careful control of gas flow ratio of C@sub 2@H@sub 2@ : SiH@sub 4@ : Ar in low pressure PIIP was needed for the growth of DLC films with optimal combinations of increased sp3 bonding structure, high hardness and density, and improved optical

properties. The resultant Si-doped DLC films with a Si content up to 28 at % exhibited a higher optical transmittance in the visible light range. The tribological tests of Si-doped DLC films were carried out using a pin-on-disk tribometer in ambient air at about 15% relative humidity. The results shown that Si-doped DLC films exhibited a low friction coefficient of 0.03-0.08 and an enhanced wear resistance despite of deposition of the films on Si (100), glass, or PMMA substrates. The effects of ion impingement during PIIP deposition on the formation of Si-incorporated DLC films were discussed.

4:20pm **TF+VM-MoA8 Surface Acoustic Wave Propagation Properties of Nitrogenated Diamond-like Carbon Films***, **J.Y. Kim**, H.J. Chung, H.J. Kim,* Seoul National University, Korea*; H.M. Cho, H.K. Yang, J.C. Park,* Korea Electronics Technology Institute

Surface acoustic wave (SAW) devices have become more important as mobile telecommunication systems need high-frequency, low-loss, and down-sized components. Higher-frequency SAW devices can be more easily realized by developing new high-SAW-velocity materials. The ZnO/diamond/Si multilayer structure is one of the most promising material components for GHz-band SAW filters because of its high SAW velocity above 10,000 m/sec. Recently, DLC films are also considered to have a potential for this application, since their physical properties, such as hardness and elastic modulus, are comparable with those of diamond. However, the residual stress during deposition is an obstacle to this application, because the film having high residual stress could not sustain during the full fabrication process of SAW devices. Recently, there have been many reports of the nitrogenated DLC films, which is mainly driven by the possibility of realizing the superhard @beta@-C@sub3@N@sub4@ phase. Some researchers reported the residual stress reduction by nitrogen incorporation without any other significant change in film mechanical properties. In this study, nitrogenated (a-C:N) films were deposited by reactive sputtering method. The a-C:N film properties were investigated using Raman spectroscopy, FT-IR, AES, and x-ray reflectivity (GIXR). To investigate the SAW propagation characteristics of the a-C:N films, SAW filters were fabricated using interdigital transducer electrodes between the ZnO layer and a-C:N/Si(100), which were used to excite surface acoustic waves. SAW velocities were calculated from the frequency-response measurements of SAW filters. A generalized SAW mode with velocities between 5,000 m/s and 7,000 m/s were observed as well as a high velocity Pseudo-SAW mode with 14,000 m/s. We also calculated the film elastic constants from SAW propagation velocities in the layered structure.

4:40pm **TF+VM-MoA9 High Deposition Rate Diamondlike Carbon Films Deposited using Permanenet Magnet Electron Cyclotron Resonance Plasmas***, **C. Doughty**, J.B. Bailey,* ASTeX PlasmaQuest

We report deposition of hard diamondlike carbon films at deposition rates exceeding 500 nm/min using permanent magnet ECR plasma CVD. These films are characterized by high hardness ~10-20 GPA and stresses ~300-500 MPa. The deposition rates obtained exceed typical values for plasma CVD deposition by a factor of 10-50 and enable a range of novel applications including economically attractive deposition of >10-um-thick films. These films have electrical resistivities ~10^12 ohm cm at 1 MV/cm, and optical bandgaps ~2 eV. Index of refraction can be controlled over the range 1.7 - 2.2 by manipulation of the deposition parameters. Film hardness has been measured by nanoindentation and will be reported as a function of deposition parameters. Adhesion promoting processes have been developed and films exceeding 5 um thickness have been deposited on silicon, glass and stainless steel substrates without delamination failures. Thin films (

5:00pm **TF+VM-MoA10 Polymerization in Remote Hydrocarbon Deposition Plasmas***, A. de Graaf, M.F.A.M. van Hest, **M.C.M. van de Sanden**, K.G.Y. Letourneur, D.C. Schram,* Eindhoven University of Technology, The Netherlands

The chemistry of expanding argon plasmas into which either methane (CH@sub 4@) or acetylene (C@sub 2@H@sub 2@) is injected for fast deposition of a-C:H, DLC and diamond films was studied by means of mass spectrometry, Fourier transform infrared absorption and in situ ellipsometry. The measurements reveal that the plasma chemistry of the expanding Ar/C@sub 2@H@sub 2@ and Ar/CH@sub 4@ plasmas is dominated by argon ion induced dissociation of the precursor gas. For acetylene injection the ion-induced dissociation is very efficient leading to complete depletion under certain conditions. For methane injection however, even under conditions of highest reactivity 100% dissociation of the precursor gas can not be reached. In an Ar/CH@sub 4@ plasma under certain conditions up to 40% of the injected precursor flow is transformed into C@sub m@H@sub n@ (m>1) polymers. In an Ar/C@sub 2@H@sub 2@ plasma the polymerization is much less (maximum 4%) and preferentially C@sub 2m@H@sub n@ polymers are formed. This suggests that in an Ar/C@sub 2@H@sub 2@ plasma the C@sub 2@H radicals are the main building blocks in the polymerization process and that they are probably also the dominant radicals in the deposition process. In the case of acetylene injection the deposition rate as measured in situ by ellipsometry is proportional to the depletion of the precursor gas. For methane injection however this proportonality does not hold even when the polymerization is taken into account. The difference in the polymerization rate and the dependence of the deposition rate on the gas depletion for the two plasmas is attributed to the different loss probabilities of the radicals formed in the dissociation. It is suggested that the large amount of C@sub 2@H@sub 2@ formed in the Ar/CH@sub 4@ plasma may lead to formation of radicals which, due to their relatively high loss probability, may become the dominant growth precursors.

Magnetic Interfaces and Nanostructures Technical Group
Room 618/619 - Session MI+VM+AS-TuM

Magnetic Recording: Media

Moderator: H.T. Hardner, Seagate Technology

8:20am **MI+VM+AS-TuM1 Perpendicular Patterned Media: Fabrication and Demonstration of Data Storage**, ***J. Wong***[1], *A. Scherer,* California Institute of Technology*; M. Todorovic, S. Schultz,* University of California, San Diego

Patterned media has been proposed as one of the solutions to extending data storage densities beyond 100Gbits/in@super 2@. We have fabricated perpendicular patterned media using a combination of high resolution electron beam lithography, dry etching, and electroplating. Futhermore, we have successfully demonstrated data storage in such structures. We first use vector scanned electron beam lithography to define the dot array pattern on the PMMA coated Al@sub 0.9@Ga@sub 0.1@As/GaAs substrate. After development, this pattern is transferred into the substrate using Chemically Assisted Ion Beam Etching (CAIBE). Immediately following CAIBE, we convert the Al@sub 0.9@Ga@sub 0.1@As layer into (Al@sub 0.9@Ga@sub 0.1@)@sub 2@O@sub 3@ using wet thermal oxidation. We take advantage of the highly selective etching properties of GaAs and the durable masking properties of (Al@sub 0.9@Ga@sub 0.1@)@sub 2@O@sub 3@ to create high aspect ratio Ni columns. After the dot arrays are defined in the substrate, we use electroplating to fill the etched holes with Ni, followed by polishing.@footnote 1@ Using Magnetic Force Microscopy, we find that the Ni columns are stable single domain magnets. We demonstrate data storage in these structures by controllably orienting the magnetization of individual 170nm diameter Ni columns using conventional thin film write poles. We subsequently read back the stored information using current MR or GMR read heads.@footnote 2@ This demonstration bridges the gap between the fabrication of such structures and their use in actual magnetic storage systems. Work is in progress to characterize higher density arrays (~1.3, 2.6, and 5.2Gbits/in@super 2@) in the form of data tracks (1µm in the x-direction and 0.5, 0.25, and 0.125µm apart respectively in the y-direction). @FootnoteText@ @footnote 1@ J. Wong et al., J. Appl. Phys. 85, 5489, 1999. @footnote 2@ M. Todorovic et al., Appl. Phys. Lett. 74, 2516, 1999.

8:40am **MI+VM+AS-TuM2 Ion Beam Patterning of Magnetic Recording Media With a Stencil Mask**, ***B.D. Terris***, *L. Folks, D. Weller, J.E.E. Baglin, A.J. Kellock,* IBM Almaden Research Center*; H. Rothuizen,* IBM Zurich Research Lab*; P. Vettiger,* IBM Zurich Research Lab, Switzerland

In conventional scaling of magnetic recording media, the grain size is reduced as the bit density is increased, while the number of grains per bit is held approximately constant to maintain signal to noise levels. This scaling approach, however, will reach a fundamental limit when the grain sizes become so small that they are subject to reversal due to thermal excitation on time scales of less than the required data retention time. One approach to circumventing this thermal limit is to create magnetic bits that behave as single magnetic entities, e.g. either single domains or a collection of strongly coupled grains, rather than the hundreds of weakly coupled grains per bit found in conventional granular recording media. In one approach to patterned media, ion beam irradiation is used to locally alter the magnetic properties of thin Co/Pt multilayer films.@footnote 1@ With sufficient ion dose, the easy axis of magnetization is rotated from out-of-plane to in-plane. We have used this process in conjunction with a silicon stencil mask having 1 micrometer diameter holes to pattern regularly spaced micrometer-sized regions of magnetically altered material over areas of a square millimeter. The nature of these magnetic structures has been investigated by magnetic force microscopy. The technique is demonstrated with mask-sample spacing as large as 0.5 mm. In addition, smaller regions of magnetic contrast, down to 100 nm, were created by using two masks with partially overlapping micrometer holes. Unlike other patterning techniques, this approach is non-contact and does not require post-processing to clean the disk, both potential manufacturing advantages. @FootnoteText@ @footnote 1@ C. Chappert et al., Science 280,1919(1998).

9:00am **MI+VM+AS-TuM3 Ion Induced Magnetization Reorientation in Co/Pt Multilayers for Patterned Media**, ***D. Weller***, *J.E.E. Baglin, K.A. Hannibal, M.F. Toney, L. Folks, A.J. Kellock, M.E. Best, B.D. Terris,* IBM Almaden Research Center

Ion beam patterning of magnetic thin films using stencil masks is a prospective path towards ultrahigh-density magnetic recording media. Co/Pt multilayers are ideally suited for this application, since they undergo a spin-reorientation transition from easy axis out-of-plane to easy axis in-plane upon irradiation with ions of suitable energy and dose.@footnote 1@ The mechanism, leading to the observed modulation in magnetic properties is of great fundamental and technological interest and will be discussed in this paper. Electron beam deposited Co/Pt multilayers with representative structure [Si-substrate/SiNx/20 nm Pt buffer/10x(0.3nm Co/1 nm Pt)/2 nm Pt cap layer] were used. These structures have high coercivity (H@sub C@ = 5000- 8500 Oe) and exhibit square perpendicular hysteresis. The high coercivity is attributed to the the large perpendicular anisotropy (K@sub u@ = 4.3 10@super 7@ erg/cm@super 3@) and granularity of these films as indicated in AFM surface topography measurements. The structures were subjected to various doses and currents of 700 keV N@super +@ ions and investigated after each irradiation step using grazing incidence X-ray reflectivity and Kerr hysteresis loop measurements. Direct evidence for ion beam mixing at the Co/Pt interface is found from the XRD data. In particular, we find an almost linear decrease of the integrated intensity of the first grazing incidence Bragg peak with ion dose. This correlates with the measured remanence ratio and anisotropy, however, not with the coercivity, which drops off much faster. @FootnoteText@ @footnote 1@ C. Chappert et al., Science 280, 1919 (1998).

9:20am **MI+VM+AS-TuM4 Texture and Strain in Cr/NiAl Films Grown on Glass Substrates**, ***G. Khanna***[2], *B.M. Clemens,* Stanford University

Glass has recently emerged as a promising candidate to replace NiP/Al in magnetic recording media due to its smooth surface and high shock resistance. A NiAl seed layer may be employed to produce the desired (112) orientation in the Cr underlayer and a (1010) orientation in the Co-alloy magnetic layer. Since NiAl forms the template for subsequent growth of Cr and Co, determining its growth texture and strain is critical to understanding the microstructure and magnetic properties of the media. We report on synchrotron radiation experiments on Cr/NiAl films of various thicknesses grown on glass substrates at elevated temperatures. Our results demonstrate that the growth of the NiAl (and consequently the Cr) on glass substrates is markedly different from Cr growth on traditional Al/NiP substrates. While a strong (002) out-of-plane texture develops at elevated temperatures in the latter case, no particular growth orientation dominates in the NiAl. Both (110) and (112) reflections appear out-of-plane in Cr films grown on thin seed layers. This result indicates that both growth orientations are present in the NiAl since the Cr grows epitaxially on the NiAl surface. Furthermore, several out-of-plane reflections appear in thick NiAl films which implies that, initially, there is simultaneous growth of NiAl grains having several different orientations. Integrated intensities of (110) reflections suggest that NiAl (110) grains are overgrown as the film thickness increases. A comparison of pole figures shows that the NiAl (110) peak is shifted to @Delta@ @psi@ = 30 from the out-of-plane direction for thicker films. The evolution of the texture with depth may be quantified using grazing incidence geometry and varying the incident angle. Intensity ratios from GIXS in-plane reflections corresponding to particular out-of-plane orientations are used to depth profile the texture. The in-plane reflections may also be used to determine the inhomogeneous strain in both layers.

9:40am **MI+VM+AS-TuM5 Ultrafast Magnetization Dynamics in Magnetic Thin Films**, ***T.M. Crawford***, Seagate Research **INVITED**

If one extrapolates the current growth trends for disc drive data rates, the data rate expected by the year 2005 is 2.4 Gbits/sec, requiring magnetization reversal frequencies in the GHz range. However, Permalloy (NiFe), a standard material used for inductive write heads, exhibits ferromagnetic resonance (FMR) at ~ 630 MHz, which is a 10%-90%, precession-limited switching time of 550 ps. While increasing the saturation magnetization and/or anisotropy shifts this resonance to higher frequencies, the gain in switching speed is proportional to only the square root of such increases. As a result, operating magnetic recording heads at or near the FMR frequency may be a necessity to achieve the desired data rates in future storage devices. This rapid increase in data rate toward the

[1] **Falicov Student Award Finalist**

[2] **Falicov Student Award Finalist**

fundamental switching speed limit has generated renewed interest in the field of high speed magnetic switching and magnetodynamics, originally studied extensively in the 1950's and 1960's. This renewal has been assisted by the availability of faster electronic and optical techniques with improved signal-to-noise for characterizing magnetic materials and devices at times well below 1 ns. Recent contributions to this field in the form of time-domain switching measurements, where the film magnetization is driven far from equilibrium, will be reviewed. Subtle material-dependent phenomena which have been observed by these techniques, including possible differences in bulk and surface magnetic properties, will be discussed, as will the possibility of actively controlling the magnetodynamics to achieve a desired behavior. Finally, the extension of these techniques to more complicated materials systems and nanoscale device structures will be addressed.

10:20am **MI+VM+AS-TuM7 Temperature Dependent Characterization of Thermal Stability of Longitudinal Magnetic Recording Media**, ***A. Moser***, *D. Weller, E. Fullerton, K. Takano,* IBM Almaden Research Center

Temperature dependent characterization of thermal stability was performed on a series of magnetic recording media at temperatures between 300 K and 420 K using a static write/read tester.@footnote 1@ The investigated samples are CoPtCr alloys with thicknesses in the range between 5.5 nm to 13 nm. First, the thermal stability of a recorded bit track was studied by measuring the time-dependence of the read-back amplitude between 0.8 s and 70000 s. Second, the time-dependent coercivity was measured by applying a magnetic field pulse of 5 ns to > 60 s width opposite to the sample's initial magnetization. Finally, the samples were characterized by SQUID magnetometry yielding temperature dependent coercivities, viscosity parameters and irreversible susceptibilities. The measurements are discussed with a quasi-independent particle model. The measured stability ratios (ratio between energy barrier for magnetization reversal to thermal energy) and signal decay rates are found to decrease faster than simple scaling with temperature would predict. @FootnoteText@ @footnote 1@ A. Moser, D. Weller and M.E. Best, J. Appl. Phys. 85, 5018 (1999)

10:40am **MI+VM+AS-TuM8 High Resolution FE-Auger Electron Spectroscopy: Applications in Magnetic Recording, Heads and Media**, ***C.A. Fenno***, Seagate Technology - Colorado Design Center

As the Disk Drive Industry pushes toward higher capacity, smaller form factors, and better performance, head and disc design has changed considerably. Technological advances have resulted in decreased dimensions; thinner layers on the disc and within the head reader element, and lower flight heights. As a result the tools used in material characterization requires improved spatial resolution, increased depth resolution and increased spectral resolution. One answer to the challenge of evaluation and characterization of smaller disc and head features is FE-Auger Electron Spectroscopy. FE-Auger provides elemental analysis with excellent spatial resolution. In the best case the electron spot size can achieve 20nm although in the practical case on disc and head features an electron spot size of 60-100nm is more typical. Features of sub-micron dimensions are routinely analyzed with FE-Auger. In some cases high spectral resolution FE-Auger data can reveal chemistry as in the case of titanium-, silicon-, and aluminum-based particles as well as in the case of some oxides and carbides. This chemical data is obtained from particular energy shifts or peak shape change from the respective materials. This presentation will show several examples where the high spatial and spectral resolution available with FE-Auger was instrumental in diagnoses in drive failure analysis.

11:00am **MI+VM+AS-TuM9 Characterization of Co/CN@sub x@ Granular Media Prepared by Nanolamination**, *C. Ruby, J. Du, R. Zhou,* ***S.C. Street***, *J. Barnard,* The University of Alabama

Cobalt-carbon thin films proposed for use as granular magnetic media are generally prepared by co-deposition sputtering. An alternative method is nanolamination of the component layers (media and matrix) followed by annealing. The potential advantages of this approach include precise control over component volume fractions and ease in fabricating large, uniform samples. We have produced and characterized thin film granular structures prepared from nanolaminate layers of Co and CN@sub x@. TEM studies of the microstructure indicate that for certain samples discrete domains are generated, with mean grain sizes of around 20 nm, with near lognormal distribution. These films have coercivities above 1200 Oe. Surface characterization by XPS depth profiling indicates that the annealed films have oxidized cobalt in the very near surface region, although initial study did not show any magnetic hysterisis loop shift. AFM measurements show that the surface of the film roughens significantly upon annealing, with the RMS roughness increasing from 0.2 nm to 1.0 nm. Thus, it appears that the mixing involved in the annealing process, which gives rise to grain formation, also degrades the smooth surface of the CN@sub x@ capping layer and exposes some of the cobalt to ambient. The implications of this process on tribological issues is explored.

11:20am **MI+VM+AS-TuM10 Characterization of Hard Disk Drives by Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS)**, ***B. Hagenhoff***, *R. Kersting,* TASCON GmbH, Germany*; D. Rading, S. Kayser, E. Niehuis,* ION-TOF GmbH, Germany

Hard disks used in hard disk drives consist of a complex inorganic and organic layer structure. Whereas substrate near layers are inorganic of origin and can be comparatively thick, layers closer to the surface become very thin and are finally covered by an organic F containing lubricant. Defective production processes as well as normal use can change the original layer structure and composition. For an analytical characterization of these changes a technique is required which gives detailed information on the chemical composition in lateral as well in depth directions. Time-of-flight secondary ion mass spectrometry (TOF-SIMS) is ideally suited to perform this task because it offers elemental as well as molecular information with high sensitivity. A 3-D representation of the sample can be obtained by combining sputter depth profiling and imaging. With modern TOF-SIMS instruments meanwhile a lateral resolution of less than 200 nm and a depth resolution of only a few nm can be obtained. We therefore applied TOF-SIMS to the characterization of commercially available hard disks. We concentrated on the identification of the lubricant present in the uppermost monolayer, screening for corrosion spots and layer structure elucidation. Special emphasis was laid on the automation of measurement and data evaluation routines in order to enhance sample throughput for industrial applications. Examples for spectroscopy, imaging and depth profiling will be presented.

Vacuum Metallurgy Division
Room 620 - Session VM+TF-TuM

Ionized Plasma and Chemical Vapor Deposition

Moderator: B. Sartwell, Naval Research Laboratory

9:00am **VM+TF-TuM3 New Plasma Sources for Ionized PVD**, ***D.N. Ruzic***, University of Illinois, Urbana*; D.B. Hayden,* Novellus Systems Inc.*; D.R. Juliano, M.M.C. Allain,* University of Illinois, Urbana **INVITED**

Three plasma sources have been investigated on a commercial magnetron sputtering system: an inductively coupled plasma (ICP) coil, a helical resonator, and an external helicon antenna.@footnote 1@ The main variables presented are the ionization fraction to the substrate, the deposition rates, the electron density and temperature. The ICP coil with an Al target achieved ionization fractions to the substrate in excess of 80%. The deposition rates are around 1500 Å/min. Electron densities are found as high as 2.6±0.3 X 10@super 11@ cm@super -3@. The main drawback to the ICP approach is that the coil is too intrusive, leaving visible shadowing effects which destroy uniformity. The coil sputters some, and also flakes off built-up deposited metal, which can contaminate the system. The helical resonator coil has a much larger diameter and avoids the shadowing effects. Ionization fractions are found with a Cu target at 73±15% under conditions with deposition rates of 1000 Å/min. The electron densities approach 2. X 10@super 12@ cm@super -3@. A ground at the center of the coil eliminates the sputtering problem by maintaining a DC bias of 0~V. There is still metal flaking off the coil as metal builds up on it. The helicon antenna sits remotely outside the vacuum system, so all shadowing and contamination problems are eliminated. Cu ionization fractions to the substrate of 51±10 % with a deposition rate of 850 Å/min. are found using one remote source. The plasma density was only 2. X 10@super 11@ cm@super -3@, but the temperature of that plasma was significantly higher than without the remote helicon present. Six or more remote sources are envisioned to sit around a sputtering chamber, which can help control uniformity while increasing the ionization further. Since there is no threat of contamination inside the vacuum chamber and the substrate to target distance can remain small, the helicon source may have the highest potential of these three secondary sources in industrial IPVD applications. @FootnoteText@ @footnote 1@ D.B. Hayden, D.R. Juliano, M.N. Neumann, M.M.C. Allain, D.N. Ruzic, "Helicon Plasma Source for Ionized PVD," Surf. Coating Tech., to be published (1999).

9:40am **VM+TF-TuM5 Simulations and Experimental Measurements of a Hollow Cathode Magnetron Ionized Metal Plasma Deposition System**, ***G.I. Font***, *K.F. Lai, Q. Lu,* Novellus Systems, Inc.*; M.J. Kushner,* University of Illinois, Urbana

The hollow cathode magnetron (HCM) is a novel new plasma source used for ionized metal deposition. The HCM employs geometric, electrostatic, and magnetic confinement to produce a high density plasma (>1e+12 #/cm3). This plasma serves as a source of ions for sputtering the target and metal ions for deposition on a wafer. In the results reported here, numerical simulations of the HCM using a copper target were performed using the Hybrid Plasma Equipment Model (HPEM) developed at the University of Illinois. The HPEM iteratively combines particle and fluid transport models for ions, electrons, and neutrals to simulate HCM performance. The model includes sputtering of the target by metal and argon ions, secondary electron emission, magnetic confinement of electrons, and thermalization and ionization of sputtered neutrals. The numerical results are compared with experimental Langmuir probe and wafer deposition profile measurements. The numerical results are found to systematically track the experimental measurements. In both experiments and modeling of an HCM, the magnetic field configuration resulted in a confined 'beam' of plasma emanating from the HCM. The physics of the operation of the HCM is described as supported by numerical and experimental results.

10:00am **VM+TF-TuM6 Low Temperature Polysilicon Deposition by Ionized Magnetron Sputtering**, ***J. Joo,*** Kunsan National University, Korea

Ionized PVD has deep potential for wide range of applications in thin film deposition. Poly Si deposition on glass should be one of them. A-Si based TFT technology has a limit of electron mobility less than 1 cm@super 2@ V/sec. Excimer laser annealing would be one solution for recrystallization but too expensive and slow process in economic point of view. As Si has very high melting temperature, the required substrate temperature for crystallization is well over the softening temperature of conventional glass in flat panel industry. RF ICP based ionized magnetron sputtering was applied to deposit polysilicon on glass substrate while keeping substrate temperature less than 400C. From X-ray diffraction analysis, small evidence for microcrystalline Si was confirmed at 250C of substrate temperature and floating substrate potential. The effects of pulsed dc sputtering power, substrate biasing frequency and ICP driving frequency will be addressed in detail.

10:20am **VM+TF-TuM7 A Study of the Mechanical Behaviour of Plasma Deposited Silica Films on Polycarbonate and Steel**, *A. Hofrichter, A. Constantinescu,* CNRS, Ecole Polytechnique, France*; S. Benayoun,* E.N.S.A.M, France*; P. Bulkin,* ***B. Drévillon,*** CNRS, Ecole Polytechnique, France

The deposition of silica for protective coatings on polymers is of increasing interest for various applications. Key issues in the mechanical behaviour of the film are the properties of the film-substrate interface that can be modified by different pretreatments. The objective of this work is to estimate the constitutive behaviour of the film and the interface by a serie of mechanical experiments and computer simulations. In order to gain a better understanding of the involved phenomena a comparison between depositions on polycarbonate and stainless steel have been performed. The films are deposited in a low pressure (1 mTorr), scaleable integrated distributed microwave 2.45 GHz electron cyclotron resonance (IDECR) reactor, which allows fast deposition at room temperature of dense, stoechiometric silica. The internal stress of the films was evaluated with profilometry and their Young modulus measured by the vibrating slab technique. Microscratch as well as nano-, micro- and Vickers indentation tests were performed on polycarbonate and steel samples for different thickness and processing powers. As indentation measurements can not be interpreted directly, the tests have been simulated by finite elements using the Castem2000 code (CEA-France). The simulated indentation curves and the final shape of the indent were compared to the measurements. The obtained stress and strain distribution in the film conducts to a reasonable explanation of the crack system observed on the indented surfaces. Finally a parametric study of the influence of the material parameters of the interlayer on the global mechanical behaviour will also be presented.

10:40am **VM+TF-TuM8 Carburizing of Tantalum by Radio-Frequency Plasma Assisted CVD**, *A. Rubinshtein,* Ben-Gurion University*;* ***A. Raveh,*** NRC-Negev, Israel*; J.E. Klemberg-Sapieha, L. Martinu,* Ecole Polytechnique, Canada

Tantalum carbide has a great potential as an alternative to tantalum and tantalum oxide for applications requiring thermal stability and corrosion resistance. In the present work we are studying hard TaC layers prepared by inductive rf plasma-assisted CVD (IPACVD) in different gas mixtures containing argon, methane, and hydrogen. The IPACVD approach combines plasma-induced diffusion with chemical vapor deposition. Maximum temperature of the tantalum substrate measured during 6 hours of processing time was 900 degC. Microstructure of the TaC layers was characterized by XRD, AFM, AES, and XPS, and the mechanical properties were studied by micro- and nanoindentation techniques. Close correlation between carburizing parameters, microstructure and mechanical properties of the layers has been established. The best mechanical performance in terms of elasto-plastic properties (microhardness of about 25 GPa) were obtained for several micrometers thick TaC films prepared at rf power levels between 1.6-2.0 kW and pressures between 40 and 60 mTorr. The effect of gas composition, rf power and substrate temperature on the layer composition (TaC/Ta@sub 2@C phase ratio), and the mechanical behavior will be presented and discussed.

11:00am **VM+TF-TuM9 Electrical and Pressure Probe Measurements of a Hollow Cathode Magnetron Plasma**, ***K.F. Lai,*** *Q. Lu, J. Chau, G.I. Font,* Novellus Systems

The hollow cathode magnetron (HCM) is a new type of high-density plasma device developed for ionized physical vapor deposition (I-PVD). While I-PVD using RF inductively coupled plasma has a plasma density of 10@super 11@ to 10@super 12@ cm@super -3@ and operates best above tens of mTorr, the HCM achieves high levels of ionization at only a few mTorr, primarily due to its extremely high plasma density (~10 @super 13@ cm@super -3@). The plasma profiles of a HCM were measured using Langmuir probes and a novel pressure probe under various operating conditions for two different target materials (Cu and Ti). With the exception of the plasma edge where the presence of an energetic electron tail was clearly evident, the electron energy distribution function (EEDF) was approximately Maxwellian. The measured plasma density was found to increase linearly with the magnetron power whereas the electron temperature only has a weak dependence. Under similar operating conditions, the Ti HCM has a plasma density ~30% higher than that of Cu. A novel pressure probe was used to measure both the argon neutral and ion density profiles. Argon neutrals were measured when the probe was biased slightly above the plasma potential whereas both the argon neutrals and ions were collected when the probe was negatively biased. The percentage of gas rarefaction was found to increase with sputtering power but was only weakly dependent on argon density. The argon ion density profile (deduced by alternating the pressure probe bias) has similar shape as the electron density (measured by the Langmuir probe) indicating that argon is the dominant ion species. The experimental results are in good agreement with simulation using the hybrid plasma equipment model (HPEM) code.

Vacuum Metallurgy Division
Room 620 - Session VM+MI+AS-TuA

Magnetic Recording: Head/Disk Interface and Overcoats

Moderator: Y.W. Chung, Northwestern University

2:00pm **VM+MI+AS-TuA1 Ultra Thin DLC Film as Magnetic Disks Overcoat**, ***X. Chu***, *B. Zhang, K.. Johnson,* MMC Tech.

Sputtered DLC film of 100 to 200 A has been used for protective coating on thin film magnetic recording disk for years. DLC overcoat material of choice is hydrogen and/or nitrogen doped amorphous carbon deposited by magnetron sputtering. Increasing areal density in magnetic hard drives requires thinner overcoat to reduce signal loss between magnetic film and read head. Functional overcoat with thickness of 50A will be needed for next generation recording medium. Tribological performance of sputtered carbon films suffers at values below 100 A because of a degradation in physical properties. Alternative deposition techniques, such as ion-beam deposition process, create denser and harder films with the improved physical and tribological properties. In this paper we will present data on the deposition and characterization of 50 A to 30 A DLC films deposited both by sputter and IBD process. Process parameter effect on structure and mechanical properties of sputtered CNx, CHx film, and ion beam deposited CHx was studied. XPS and Raman were used to characterize film microstructure and showed the sputtered CNx film was mostly sp2 bonded. Tribology of the films were tested by Contact Start Stop (CSS) testers and the result of carbon wear can be correlated to AFM nano-wear test. 30A ion-beam deposited CHx film showed good CSS tribological performance comparable to 100 A sputtered films.

2:20pm **VM+MI+AS-TuA2 Tribological Properties of Protective Carbon Coatings Used in Magnetic Storage Devices Investigated on a Sub-Nanometer Scale**, ***A. Wienss,*** University of Saarbrücken, Germany*; G. Persch-Schuy,* IBM Germany Storage Systems Division, Germany*; U. Hartmann,* University of Saarbrücken, Germany

Ultrathin carbon coatings are used in the magnetic storage industry to protect sensitive sensor heads and magnetic media against mechanical damage. Such a damage can be modelled by artificially generated scratches using Scanning Force Microscope (SFM) techniques. Loading forces in the µN range are applied, resulting in scratches with residual depths of only a few Å. A special image subtraction technique is presented which allows careful analysis of tiny grooves even on rough surfaces. This technique compensates for drift effects during scanning. The scratching resistance of various a-C:H and CN@sub x@ films is determined. For a-C:H, an increasing amount of hydrogen results in a decreasing scratching resistance, which is a well-known behavior. Beyond a certain hydrogen content, a further increased hydrogenation causes a reproducible, slight increase of the scratching resistance. In order to explain this, the role of the friction coefficient will be discussed.

2:40pm **VM+MI+AS-TuA3 Ultrathin Overcoats For Magnetic Media: Is Hardness What We Are Looking For ?**, ***B. Marchon,*** IBM Almaden Research Center **INVITED**

As areal recording densities approach 20 Gigabit per square inch, the demand for ultrathin media overcoat (<5 nm) becomes more pressing. This talk will attempt to identify the various properties that are required to achieve good performance under increasingly severe mechanical and environmental conditions. In an attempt to bridge the process-performance gap, we will review the details of the head/disk interface system, and how the mechanical and chemical structure/properties can be optimized to achieve the required reliability. In particular, issues related to tribochemistry and interactions with the lubricant will be addressed, as well as a general discussion on what specific mechanical properties are important.

3:20pm **VM+MI+AS-TuA5 Air Bearing Collision Dynamics**, ***S.E. Stupp,*** *R.J. Blanco, T. Riener, B.D. Strom,* Quantum Corporation **INVITED**

A few years ago, a disk drive program encountered an unusual problem: drives built with one vendor's heads (vendor A) suffered from an excessive number of thermal asperity events (TAs); drives built with head's from another vendor (vendor B) did not have as many TAs, but they did have a number of crashes. Spinstand experiments confirmed that there was a significant difference in the response of the two vendor's air bearings to collision with the 100 nm high asperities found in these drives. For example, the fly height of vendor A's heads was essentially unchanged on contact with an asperity, while vendor B's heads exhibited a fly height change (these differences may explain the drive results). The underlying problem in the drive program was ultimately traced to particulate contamination and was corrected. However, the experiments raise an interesting question: Why is there a difference in the dynamic response of the two vendor's heads? In this work we present a systematic study of this problem, which we christen Air Bearing Collision Dynamics (ABCD). The asperity collision responses of several different air bearing designs (including full rail and island type) were studied by measuring the TA signal, the change in flying height, acoustic emission (AE), and laser Doppler vibrometry, in controlled experiments on a spinstand. Since a large enough asperity can cause any head to crash, the asperity size was modest (approximately equal to the fly height). In agreement with our earlier experiments, differences in the response of the different air bearings were found. In addition, the AE signals indicate that certain island type air bearing designs can undergo multiple head-disk contacts after the asperity has passed. These results are potentially concerning, because many head vendors are moving towards this type of air bearing design. Finally, in an attempt to understand the origin of the differences in the dynamic response of different air bearing designs, we report the results of numerical modeling of the asperity and air bearing designs.

4:00pm **VM+MI+AS-TuA7 Interaction of Fluoroalcohols and Fluoroethers with Various Types of Carbon Overcoats**, ***N. Shukla,*** *A.J. Gellman,* Carnegie Mellon University

This work is focussed on understanding the fundamentals of head-disk interface tribology at very low flying heights and higher spin rates. Since there will be room for only a single molecular monolayer of the lubricant on the disk surface at low flying heights we have studied the molecular level interaction of lubricants with carbon overcoats that protect the disk surface. We have modeled a most commonly used PFPE lubricant (Fomblin Zdol) using short chain model compounds and measured the desorption energy of these compounds on carbon films. The short chain model compounds used are 2,2,2 trifluoroethanol (CF@sub3@CH@sub2@OH) and perfluorodiethyl ether (CF@sub3@CF@sub2@OCF@sub2@CF@sub3@) which are representative of both the end group and the main chain of Fomblin Zdol. Temperature programmed desorption spectroscopy is used to measure the desorption energy of model compounds and also to understand the nature of the interaction of these short chain compounds with carbon overcoats. Initial results show that ethers interact with carbon overcoats through electron donation from the oxygen lone pair electron and the alcohols interact with carbon overcoats through hydrogen bonding. In addition we have studied the effect of various film compositions on the interaction of the lubricants in order to understand if the film composition has any effect on the nature of the bonding of the lubricant. The different types of overcoats used are hydrogenated, nitrogenated, diamond-like carbon and Ion beam sputtered overcoats. We have observed that by varying the percentage of hydrogen or nitrogen content in the film composition or by changing the carbon overcoat deposition conditions as in diamond like carbon overcoats or in an ion beam sputtered overcoat there is no significant change in the interaction of the lubricant with carbon overcoat. The alcohols however bond stronger than ethers on all overcoats, which is consistent with the structures, proposed by diffusion measurements.

4:20pm **VM+MI+AS-TuA8 Angle Resolved ESCA Methods: Molecular Conformation of Fluorocarbon Lubricant**, ***M.A. Karplus,*** *D.J. Pocker,* IBM-SSD

Simple but useful methods for interpreting angle-resolved ESCA data from real-world samples are presented. First is a model allowing thinly-covered and thickly-covered substrate. It can be usefully applied, even in a manual fashion, with common office spreadsheets. Next, a simple slab model, with the help of canned minimization routines, can bring out subtleties in overlayer structures. Even a deeply buried monatomic layer can be isolated. The following are presented as examples. Perfluoropolyether (Zdol) lubricant on hard disk carbon overcoat shows coexisting monolayer and multilayer regions, consistent with structures identified by surface energy@footnote 1@ and ellipsometric surface diffusion@footnote 2@ measurements. Next, layering within the lubricant shows significant perfluoropolyether backbone adjacent to the overcoat surface accompanied by a discernible excess of ether oxygen. The latter facts indicate that the inverted-U conformation sometimes sketched for bonded lubricants is an exaggeration, certainly for the system studied. @FootnoteText@ @footnote 1@ G. W. Tyndall, R. J. Waltman, and D. J. Pocker, Langmuir 14, 7527 (1998). @footnote 2@ X. Ma, J. Gui, L. Smoliar,

K. Grannen, B. Marchon, M. S. Jhon, C. L. Bauer, J. Chem. Phys. 110, 3129 (1999).

4:40pm **VM+MI+AS-TuA9 Airbearing Designs for High Density Recording**, **INVITED**

Vacuum Metallurgy Division
Room 4C - Session VM-TuP

Poster Session

VM-TuP1 Pattern Writing by Implantation in a Large-scale PSII System with Planar Inductively Coupled Plasma Source, ***L. Wu***, *D.M. Manos, T.J. Venhaus,* College of William and Mary

A large-scale PSII system has been built. With chamber 28.5in in diameter and 20in tall, pulses of up to 100KV, and base pressure in the 10@super -8@torr range, it is one of the largest PSII systems. It has been operated with hot filament, hollow cathode and recently with 22.5-inch diameter quartz window for planar RF ICP. This paper compares implantation with these plasma sources, demonstrating the advantages of RFI. It also reports measurements of the plasma density and spatial distribution using Langmuir probe for different RF power, gas pressure and plasma compositions for implanting alloys. Results of implanting alloys including large-area stainless steel cathodes to reduce field emission are reported. Metallic and semiconductor samples have also been implanted through masks with various plasma compositions to produce small geometric patterns of interest for device manufacturing. The samples are characterized by variable-angle spectrometric ellipsometry (VASE), SEM, AES, SIMS, and XPS, and for electrical and mechanical properties. Depth profiles obtained by VASE, SIMS, AES and XPS are compared to Monte-Carlo calculations (Tri-Dyn, Trim, ProfileCode). Measured lateral and depth profiles are compared to the mask features to assess lateral diffusion, pattern transfer fidelity, and wall-effects on the depth profile. The paper also presents the results of MC-hybrid and PIC calculations of the flux and angle of ion trajectories through the boundary layer predicting the uniformity of flux as a function of 3-D location on objects in the expanding sheath and to evaluate the fidelity of pattern transfer as a function of feature size. Sample heating and diffusion effect is included.

VM-TuP2 Growth of SiC Thin Films on Graphite for Oxidation Protective Coating, ***J.-H. Boo***, *M.C. Kim, C.H. Heo, S.-B. Lee, S.-J. Park, J.-G. Han,* SungKyunKwan University, Korea

We have deposited thick SiC thin films on graphite substrates in the temperature range of 700 - 850 @super o@C using single molecular precursors by both thermal MOCVD and PEMOCVD methods for oxidation protection wear and tribological coating. Two organosilicon compounds such as diethylmethylsilane (DEMS), (Et)@sub 2@SiH(CH@sub 3@), and hexamethyldisilane (HMDS), (CH@sub 3@)@sub 3@Si-Si(CH@sub 3@)@sub 3@, were utilized as single source precursors, and hydrogen and Ar were used as a bubbler and carrier gas. Highly oriented polycrystalline cubic SiC layer in [110] direction was successfully deposited on graphite at temperature as low as 800 @super o@C with HMDS by PEMOCVD. In the case of thermal MOCVD, on the other hand, only amorphous SiC layers were obtained with either HMDS or DEMS at 850 @super o@C. From this experiment, we confirmed that PEMOCVD was highly effective process in improving the characteristics of the SiC layer properties compared to those grown by thermal MOCVD. The as-grown samples were characterized in situ with OES and RGA and ex situ with XRD, XPS, and SEM. The mechanical and oxidation-resistant properties have been checked. The optimum SiC film was obtained at 850 @super o@C and RF power of 200 W. The maximum deposition rate and microhardness are 2 µmm/h and 4,336 kg/mm@super 2@ Hv, respectively. The hardness was strongly influenced with the stoichiometry of SiC protective layers. Ar-plasma pre-treatment enhanced the hardness and adhesion between SiC layer and graphite substrate due to a nucleation effect.

VM-TuP3 Novel Technique for Low Temperature Chemical Vapor Deposition of Titanium Thin Films on Mild-Steel Surfaces for Corrosion Resistance, ***J.H. Hendricks***, *M.I. Aquino, M.R. Zachariah,* National Institute of Standards and Technology

A novel, low temperature technique for growing titanium films on mild-steel substrates has been demonstrated. This method involves the use of a low pressure (600Pa) co-flow diffusion reactor in which sodium vapor and gas-phase titanium tetrachloride react in the presence of a non-reactive gas, Ar. The reaction chemistry is described by the following equation: 4Na(g) +TiCl@sub 4@(g) --@super Ar(g)@-->Ti(s)+ 4NaCl(g). In this reaction, a gas-phase alkali metal (Na) strips multiple halogen atoms (Cl) from a gas-phase metal halide (TiCl@sub 4@). This allows free Ti atoms to attach to a substrate surface placed within the reaction zone, resulting in the growth of a solid metal film. Previously, we have used this technique to grow Ti and TiN thin films on Cu substrates@footnote 1@ at 610°C and TiO@sub 2@ thin films on Si substrates@footnote 2@ at 600°C. This chemistry should be generic for the deposition of a wide class of metallic and ceramic thin films at deposition temperatures which are significantly lower than conventional techniques, and this technique could potentially be used to grow hard and superhard coatings such as CN and BN. Thermodynamics modeling was used to simulate the reactant concentrations and substrate temperatures at which the salt by-product remains in the gas-phase. The modeling predictions were compared to the experimental results and found to be in good agreement. Using the described technique, we have produced Ti thin films on mild-steel substrates with substrate temperatures of 400°C to 800°C. These temperatures are considerably lower than conventional CVD of Ti which involves the thermal decomposition of titanium tetraiodide at 1000°C to 1200°C.@footnote 3@ Lowering the temperature for Ti deposition on mild-steel is of significance since mild-steel undergoes a phase transition at 723°C.@footnote 4@ The corrosion resistance of the titanium coated mild-steel substrates were evaluated to determine the optimum substrate deposition temperature. The quality and composition of the thin films were analyzed by scanning electron microscopy (SEM), energy dispersive x-ray spectrometry (EDS), and x-ray diffraction (XRD). @FootnoteText@ @footnote 1@J. H. Hendricks, M. I. Aquino, J. E. Maslar, and M. R. Zachariah, Chem. Mater. 1998, 10, 2221-2229. @footnote 2@J. H. Hendricks, M. I. Aquino, J. E. Maslar, and M. R. Zachariah, Material Research Society Proceedings, Nov.30-Dec. 4, 1998, Boston, MA. @footnote 3@M. G. Hocking, V. Vasantasree, and P. S. Sidky, Metallic and Ceramic Coatings: Production, High-Temperature Properties and Applications, John Wiley and Sons: New York, 1989, p. 103. @footnote 4@J. M. Camp and C. B. Francis, The Making Shaping and Treating of Steel, United States Steel Company: Pittsburgh, PA, 1951, p. 1203.

VM-TuP5 Friction Evaluation and Development of Vacuum Materials for Tribological System, ***M. Tosa***, *A. Kasahara, Y.S. Kim, K. Yoshihara,* National Research Institute for Metals, Japan

Vacuum materials for movement system requires small friction as well as low outgassing. Friction and outgassing strongly depends on the surface conditions and structures of the materials. It is therefore important to evaluate fiction accurately in-situ in a vacuum as controlling such surface layer structures as contaminates layer, adsorbed layer and oxide layer by changing load and vacuum pressure. We have developed a vacuum friction measurement apparatus to evaluate sliding friction under changing the load from 1N to 0.98 mN and under the vacuum pressure from 10@super5@ Pa to 10@super-8@ Pa. Two strain gauges measure the friction force occurred at the loaded pin on the substrate sheet. The measurement was carried out on the sheets for such vacuum materials as type 304 stainless steel, copper, hexagonal boron nitride (h-BN) segregated copper film on the stainless steel, sintered h-BN plate and titanium nitride (TiN) coated stainless steel. All steel sheets are polished mechanically with diamond powder of 0.3µm in grain size. Co-sputtering deposition of h-BN chips and a copper disc target prepared segregated h-BN film. Titanium nitride was coated on the stainless steel with magnetron sputtering deposition. The result of the measurement in decreasing the vacuum pressure shows that the friction coefficient of h-BN surface segregated copper film on steel keeps about 0.1, which is smaller than any other coefficient. The friction coefficient of copper and TiN coated steel decreased gradually but still larger than that of h-BN surface segregated copper film and the friction coefficient of stainless steel increases very much in decreasing the pressure. The result of the measurement in decreasing the load shows that the friction coefficient of h-BN surface segregated copper film on steel keeps smaller than that of copper and steel sheet. Hexagonal boron nitride segregated copper film can be therefore a good candidate material for vacuum tr! ibological system.

Vacuum Metallurgy Division
Room 620 - Session VM-WeM

Advanced Surface Treatments and Coatings
Moderator: A. Inspektor, Kennametal Inc.

8:20am **VM-WeM1 Microstructural and Surface Morphological Evolution at the Atomic Scale during the Growth of Polycrystalline TiN: a TEM, XRD, HT-STM, and Modeling Study**, ***I. Petrov***, *S. Kodambaka, P. Desjardins, A. Vailionis, V. Petrova, J.E. Greene,* University of Illinois, Urbana*; L. Hultman,* Linköping University, Sweden*; G. Gilmer,* Lucent **INVITED**

TiN is widely used as a diffusion barrier in microelectronics, as a hard wear resistant coating on cutting tools, and as a corrosion and abrasion resistant layer on optical components. Even though its diffusion barrier and elastic properties are known to be extremely anisotropic, little is known regarding the mechanisms and reaction paths leading to the development of preferred orientation in polycrystalline TiN layers deposited by PVD. We have used in-situ temperature-dependent STM measurements during deposition and post-annealing, detailed post-deposition microstructural analyses, and modeling to provide atomic-scale insights into microstructural and surface morphological evolution during TiN film growth. The results show that TiN layers grown at low temperatures (@<=@ 450°C, T@sub s@/T@sub m@ = 0.20) exhibit competitive texture evolution with a columnar 111 "kinetically-limited" texture eventually becoming dominant. The columns are narrow with inter- and intracolumnar porosity and facetted surfaces. Higher growth temperatures or the use of high incident N@super +@@sub 2@/Ti flux ratios (> 5) with low ion energies (20 eV) result in non-competitive growth with the development of a fully dense essentially complete 002 preferred orientation from the initial monolayer. The above microstructural results can be understood qualitatively assuming that the activation energy E@sub s@ for surface diffusion and the Ehrlich barrier E@sub b@ at descending step edges are larger on 111 surfaces than on 002. Using this, together with the assumption that pseudomorphic forces (i.e., local epitaxy) dominate once island orientation is determined locally, the general features observed in the experiments outlined above can be replicated using kinetic Monte Carlo simulations. The in-situ STM observations of the dynamics of island growth and decay also provide important additional insights into the atomic-scale growth of TiN and related transition-metal nitrides.

9:00am **VM-WeM3 High Refractive Index -Textured Cubic Zirconia Formed in Nanolaminate Coatings Using Titania Interruption Layers**, ***J.D. DeLoach***, University of Wisconsin, Milwaukee*; C.R. Aita,* University of Wisconsin, Milwaukee, US

Thermodynamic phase evolution of bulk zirconia cooled from the liquidus (2680 @super o@C) is as follows: cubic (2360 @super o@C) to tetragonal (~1075 @super o@C) to monoclinic (STP phase). However, the pseudobinary zirconia-yttria phase diagram shows that the cubic phase is stabilized at room temperature by adding approximately 10 mole % yttria. Therefore, most reports of cubic zirconia formation in coatings of practical thickness involve yttria-doped zirconia. Undoped zirconia single layer film studies report the transformation from cubic to monoclinic zirconia with increasing crystallite size, suggesting that a finite crystal size effect aids in cubic zirconia stabilization. For this reason, zirconia phase control is easier in a multilayer scheme, by the introduction of growth interruption layers. This approach was taken by several investigators who grew cubic zirconia in zirconia/yttria nanolaminates. In these cases, cubic zirconia was formed by heteroepitaxy with either -texture cubic yttria or a -texture interfacial cubic zirconia-yttria alloy. The drawbacks of using yttria for growth interruption layers were: (1) the refractive index of the nanolaminate was considerably lowered by the presence of yttria, and (2) the -texture of cubic zirconia was stabilized by heteroepitaxy, precluding a -texture desirable for certain applications. In this study, a sputter-deposited nanolaminate structure consisting of polycrystalline zirconia-vitreous titania bilayers was used to fabricate high refractive index, undoped cubic zirconia coatings with a strong crystallographic texture. Titania was chosen as the interruption layer material because: (1) its vitreous nature encouraged low surface energy {200} cubic zirconia planes to preferentially orient parallel to the growth interface, resulting in a -texture, and (2) its high refractive index resulted in a nanolaminate with an overall refractive index of 2.23.

9:20am **VM-WeM4 Tribological Performance and Initial Finite Element Modeling of Reactively Sputtered Single and Multi-layer Chromium Nitride Thin Films**, ***S.L. Rohde***, *D. Mihut, S. Kirkpatrick,* University of Nebraska, Lincoln

This work examines the tribological properties of CrN and Cr@sub 2@N thin films both as single-layers and in a number of different multilayer structures combining CrN, Cr@sub 2@N and Cr. The overall program goal was to test the feasibility of "building-in" load support, by alternating hard/soft film layers to optimize performance on hard materials, such as hardened tools steels, as well on compliant materials like Al-alloys. The first phase of this program centers on the deposition and evaluation of an array of coating structures on various substrates. Thin films were deposited in a mixed Ar-N@sub 2@ discharge, using a single unbalanced magnetron cathode with a Cr-target operating in a metallic-mode. Three differing substrate materials were coated; 52100 bearing steel, A2 tool steel and 2024-alloy aluminum. The adhesion of the thin film/substrate couples was evaluated using scratch adhesion and Rockwell C indentation testing. Scratch adhesion values were found to range from 2 to 8 kg depending on the hardness of the substrate material; with the lowest values occurring on the most compliant substrates (i.e. 2024 Al). The wear behavior was assessed using pin-on-disk and high frequency Cameron-Plint testing. The pin-on-disk tests were performed without additional lubrication at 40 to 50% humidity using alumina and/or tungsten carbide balls as the pin materials. . The wear was reduced in most cases, with the multi-layered structures performing best on all substrate materials. The lubricated Cameron-Plint tests run on coated 52100 steel substrates favored the Cr@sub 2@N-based films, although the hardness of these multilayers is frequently lower than the corresponding CrN-based structures. While on the A2 tool steel substrates, the hardest thin film structures yielded the best PoD wear performance, these same films did not perform as well on the more compliant Al-substrates. For the Al-substrates, neither the stiffest nor the most compliant films excelled, instead multi-layer film with alternating hard/soft structures designed to provide a more graded compliance from the substrate up to the rigid top layer yielded the best results. In this case, wear rates were reduced by as much as much as three orders of magnitudes over uncoated 2024 Al. Finite element modeling studies have been initiated to try to understand the behavior of these multilayered coating/substrate combinations under specific loading conditions. While still in it's earliest stages, the ultimate goal of the modeling program will be to facilitate design and optimization of application specific coating structures into the original component design stage. To accomplish this models are being developed for previously tested film structures on various substrates, and these models will then be used to guide the development of second generation coatings, that will be used to verify and improve the efficacy of the models.

9:40am **VM-WeM5 Protective Coatings for Extreme Environments**, ***H.W. Holleck***, Forschungszentrum and Universität Karlsruhe, Germany **INVITED**

Physical Vapor Deposition (PVD) allows multifunctional protective coatings to be achieved for extreme and very complex loading situations. Among various thin-film concepts such as superhard coatings, gradient coatings, composite coatings, superlattice coatings, solid-solution coatings, metastable multifunctional coatings, nanoscale coatings, designed to improve the performance of protective coatings, the nanostructured multilayer films, the nanocrystalline composite films, and the metastable vapor-quenched compound films are able in particular to meet extreme requirements. Knowledge of specific design principles has developed over the past few years, thus adding a scientific design basis to what used to be empirical film development. Properties and functions simultaneously attainable include high levels of hardness, toughness, oxidation and corrosion resistance, abrasion and erosion resistance, as well as low friction and low wear in many applications. Multilayer coatings combining metallic hard materials e.g. TiC and TiN or TiAlN, metallic and covalent hard materials e.g. TiC and C or SiC, as well as combinations of hard and soft materials, e.g. TiN and MoS2 or C, will be discussed. Functional and structural contributions are responsible for the changes in properties produced by different numbers of single layers. Relations similar to those applying to multilayer coatings can be found for nanocrystalline films, with the grain size being the most important parameter besides phase composition. An almost unlimited source of new multifunctional coatings meeting extreme requirements are vapor-quenched multicomponent films combining materials of different hard material groups in a homogeneous film material optimized with respect to composition and structure. Examples to be discussed are TiC,TiN, TiB2, and CrN combined with compounds such as BN,B4C, SiC, and AlN. Thermodynamic and kinetic

modeling of the coating system and the deposition process allows the constitution, properties and performance of these coatings to be tailored within broad limits.

10:20am **VM-WeM7 Interface Engineering in a Combined Arc/ UBM Deposition System during Growth of Ti@sub 0.5@Al@sub 0.5@N Films on Steel: Effects of Substrate Ion Etching**, ***C. Schönjahn***, *L.A. Donohue, D.B. Lewis, W.-D. Münz,* Sheffield Hallam University, UK*; R.D. Twesten, I. Petrov,* University of Illinois, Urbana

In-situ substrate cleaning by ion etching prior to deposition in PVD processes is a key step in achieving good film adhesion, which is essential for all coating applications. Irradiation with metal or gas ions alters substrate surface chemistry, topography and microstructure thus affecting subsequent film growth. This study compares Ti@sub 0.5@Al@sub 0.5@N/ steel interfaces formed after Cr- metal ion bombardment at negative substrate biases, Us, ranging from 600 to 1200 V during a Cr cathodic arc, stabilized with a 0.06 Pa Ar background pressure. Samples pretreated with a 1200 V Ar glow discharge at a pressure of 0.6 Pa were also investigated. Microstructure and microchemistry of the interfaces was studied by XTEM samples using STEM-EDX analysis. Cr ion etching with Us = 1200 V resulted in a net removal of over 100 nm of substrate material with the formation, through implantation and radiation-enhanced diffusion, of a Cr-enriched near-surface region extending to a depth of ~10 nm. As Us was reduced to 600 V, Cr accumulated at the surface as an ~5 nm thick layer. Ar was incorporated at the surface to levels of 4 and 5 at % during Cr arc-etching and Ar glow discharge, respectively. Microstructure of Ti@sub 0.5@Al@sub 0.5@N overlayers was dramatically affected by pre-treatment procedures. Ar sputter cleaned steel surfaces (Us = 1200 V) promote nucleation of randomly oriented grains leading to a competitive column growth with small column size and open boundaries. In contrast, Cr irradiation at the same bias voltage results in local epitaxial growth of Ti@sub 0.5@Al@sub 0.5@N on steel, which is expected to improve film/substrate adhesion. Coatings deposited after Cr treatment with Us = 600V still exhibit small areas of oriented growth but porosity is also present.

10:40am **VM-WeM8 Plasma Treatment of Polycarbonate for Improved Adhesion**, ***A. Hofrichter***, *P. Bulkin, B. Drévillon,* CNRS Ecole Polytechnique, France

The deposition of silicon alloys for protective and optical coatings on polymers is of increasing interest. The understanding of the plasma polymer interaction is of prime importance here. In this work we report a study of the influence of various plasma-treatments on polycarbonate (PC) in order to clarify the adhesion mechanisms of plasma deposited silica films. Treatments and depositions were carried out in a low pressure (1 mTorr range) integrated distributed 2.45 GHz microwave electron cyclotron resonance reactor, which allows the deposition of dense stoechiometric silica at room temperature. The plasma-polymer interaction is studied by in-situ spectroscopic ellipsometry from near UV to infrared and ex situ Raman spectroscopy and atomic force microscopy. The influence of process parameters, such as power and gas composition is studied. To get better insight into the plasma modification mechanisms thin polymer layers, spincoated onto various substrates were used. This allows a precise determination of the absorption properties and the thickness evolution of the plasma modified surface layer by UV-visible ellipsometry. The absorption in the PC layer was found to increase in the UV and is attributed to the different reaction products of photo-Fries rearrangements. Particular attention was taken to evaluate the contribution of the VUV photons. The chain scission and crosslinking of pure and commercial grade polycarbonate is studied by gel permeation chromatography and correlated to the ellipsometric results. Finally we discuss the influence of the treatement on the silica adhesion, evaluated by microscratch test measurements.

11:00am **VM-WeM9 The Effects of Electron Beam Assistance on the Properties of Ion Beam Deposited CN Thin Films**, ***Y.H. Kim***, *D.Y. Lee, I.K. Kim, H.K. Baik,* Yonsei University, Korea

For the direct ion beam deposition process, the kinetic energy of ion beam have been considered as a main factor for the determination of the properties of resulting thin films. But we have investigated the effects of electron beam assistance on the properties of thin film during the ion beam deposition process of CN material. For the verification of the charge-enhanced chemical bonding, the properties of carbon nitride thin films deposited with and without electron beam assistance are compared. For the direct ion beam deposition process, negative carbon ion beam and positive nitrogen ion beam was used simultaneously. Total negative beam including negative carbon ion and electron can be emitted from the Cs ion bombardment on the graphite target. The filtering of electrons from the total negative beam by the transverse magnetic field is possible and the pure negative carbon ion beam was deposited with positive nitrogen ion beam for the less charge-enhanced chemical bonding process. The properties of DLC and CN thin films were discussed respectively by comparing the thin film growth with and without electron beam assistance. The properties of thin films were characterized by Raman spectroscopy, AES and XPS.

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