Sunday Evening
May 10, 2009
Plenary Address
In a high level seminar arranged in September 2008 by the Swedish-American Chamber of Commerce, discussions about the rapid increase of carbon dioxide content in the air took place. Some delegates compared the situation with writing a book and suggested that chapter one was ready. Chapter one dealt with the problem of getting the world to understand that the carbon dioxide increase is a problem. The delegates were in agreement that the problem was understood. It is now necessary to find the best solutions and act. The future will show if this book turned out to be a drama, a thriller or, if we are lucky, a comedy. To succeed in this we cannot just act as we have always done by improving the existing solutions. We need to have a new type of wild, daring and humanitarian solution, but still cost-efficient. Innovative solutions must be found. Any idea/solution initially has to be “tested” intellectually against the Physical Laws. Few politicians know the facts about different renewable energies. The facts are for average power: sun – 100W/m², wind (11m/s) – 1kW/m², wave (Sweden) = 5-10Kw/m wave, wave (USA) – 15-60kW/m wave and underwater current (2m/s) – 4kW/m². Another interesting fact is that the number of full load hours differs widely as follows: sun – 1,000 h/year, wind (Sweden) – 2,200 h/year, wave (Sweden) – 3,000 to 4,000 h/year, wave (USA) – 3,000 to 6,000 h/year and underwater currents – 7,000 h/year. Having these facts as background three new ways of extracting renewable energy have been invented in Sweden. For wind energy there is a solution that uses a vertical arrangement, which of course, will lead to better cost performance and lower risk for failures or breakdown. For wave energy, the solution is based on a Wave Energy Converter (WEC) placed on the sea bed (well protected) having a point absorber driving a linear generator. Arrays of WECs are then connected via underwater switchgears to the grid. For underwater current, the solution is based on a vertical arranged turbine with a slow speed generator. This will also result in a minimum or no negative influence on the environment. The trend we see nowadays is an increased focus on environment friendly solutions that will get even stronger in the future. However, it is important to question solutions that are not cost-effective and do not focus on the environment for the whole supply chain including recycling.
Keynote Address
Over 40 years of thin film process innovations have helped enable the IC industry today to produce well over $10^{18}$ transistors per year at costs of nanodollars per transistor, thereby empowering the information age. Likewise, large area thin film manufacturing has dramatically improved the performance and cost of low cost displays over the past 15 years, enabling high definition video from the handheld to the wall-mounted HDTV. The overwhelming societal and market pull today for new solutions in the field of clean energy offers an exciting opportunity to build on a similar base of technology. Through a combination of materials innovation and highly productive processing platforms we have the potential to enable new solutions for conservation, conversion and storage and thus profoundly change the economics of clean energy.
High Power Impulse Magnetron Sputtering
HIPIMS
9:30 a.m. HP-1 PVD Processes in High Aspect Ratio Features by HIPIMS

J. Weichart, M. Elghazzali, and S. Kadlec, OC Oerlikon Balzers AG, Balzers, Principality of Liechtenstein; and A.P. Ehiasarian, Materials and Engineering Research Institute, Sheffield Hallam University, Sheffield, United Kingdom

Invited 40 min. Talk

Highly ionized sputtering (HIPIMS) has been developed on rotating PVD magnetron sources for uniform deposition on 200mm as well as on 300mm single wafer tools for through silicon via (TSV) metallization in 3-dimensional packaging. The big advantage of HIPIMS is that the technology can be applied to regular PVD sources working at low target to substrate distance and by this providing a high transfer factor and a cost-effective process. The ionization degree and distribution of the plasma has been analyzed by atomic absorption spectroscopy, time and energy resolved mass spectroscopy and other methods. Processes have been optimized for Ti, Ta and Cu with RF bias applied on the substrate to accelerate the generated ions into deep silicon etched trenches and vias of very high aspect ratio (AR) up to 30:1 with vertical sidewalls. For Ti a bottom coverage of more than 20% in trenches with AR 10:1 and still more than 7% in trenches with AR 30:1 have been achieved at deposition rates of more than 28nm/s. Detailed studies in vias with scalloped sidewalls show the limitations of the ionized PVD process, the experiments are supported by Monte-Carlo-simulations of the deposition.

10:10 a.m. HP-2 Effect of the High Ion Irradiation on the Structure, Tribological and High Temperature Performance of CrAlYN/CrN Nanoscale Multilayer Coatings Deposited by the HIPIMS Technology

P. Hovsepian, A.P. Ehiasarian, and Y. Purandare, Sheffield Hallam University, Sheffield, United Kingdom; R. Braun, DLR-German Aerospace Center, Cologne, Germany; and I.A. Ross, University of Sheffield, Sheffield, United Kingdom

The high degree of ionisation and high energy of the sputtered particles in the High Power Impulse Magnetron Sputtering Discharge (HIPIMS) provides excellent conditions for deposition of highly dense and very smooth coatings in particular due to the irradiation effects. These qualities were exploited in the newly developed nanoscale multilayer CrAlYN/CrN designed to meet the demands for highly oxidation resistant and wear resistant coatings. The coatings were produced by HIPIMS/HIPIMS technology where HIPIMS was employed both for adhesion enhancement surface pre treatment as well as for coating deposition. Cross Section Transmission Microscopy revealed extremely sharp interfaces between the individual layers in the nanolaminated material and almost no layer waviness. It was found that the coefficient of friction at room temperature was not affected strongly by the residual stress and retained low values of $\mu=0.56$. High temperature tribometry revealed that after initial increase of the coefficient of friction to $\mu=0.7$ at $120^\circ$C, the value drops to as low as $\mu=0.47$ for test temperature of $650^\circ$C. At this temperature, extremely low wear coefficient of $K_c=1.8\times10^{-7}$m$^3N^{-1}m^{-1}$ was measured. Quasi-isothermal oxidation tests carried out at $850^\circ$C for exposure time of 1000 hours further confirmed the excellent high temperature behaviour of the coatings. In these conditions, HIPIMS/HIPIMS coatings showed by factor of 2 and 6 lower mass gain as compared to the UBM deposited coatings and uncoated TiAl substrate respectively.
Monday, May 11

10:30 a.m. HP-3 Industrial Impact of HIPIMS+ Technology for Chromium Nitride Coatings


Chromium Nitride (CrN) is widely used for tribological and tool applications due to its toughness, abrasion resistance and chemical inertness. For tribological applications, it plays an important role in reducing fatigue and sliding wear. It is also used as an intermediate support layer for Diamond-like Carbon (DLC) coatings. For tools applications, it is used to resist cold welding and plastic adhesion as well as to provide impact fatigue resistance. The application determines which physical properties are needed in the CrN layer. High Power Impulse Magnetron Sputtering Plus (HIPIMS+) is a deposition technology in which a significant portion of the sputtered material is ionized. This offers many advantages with regards to the process window for depositing CrN. A comparative analysis of HIPIMS+ and closed field Unbalanced Magnetron Sputtering (UBM) coatings including x-ray diffraction results and mechanical properties will be presented. The potential advantages of HIPIMS+ deposited CrN coatings for tool and tribological applications will also be discussed.

Monday, May 11

10:50 a.m. HP-4 HIPIMS-MPP Deposited Ta and Cr Coatings for High Temperature Wear and Erosion Applications

S.L. Lee, F. Yee, M. Cipollo, and S. Smith, U.S. Army ARDEC-Becht Laboratories, Watervliet, NY; and R. Chistyakov and B. Abraham, Zond Inc./Zpulser LLC, Mansfield, MA

HIPIMS-MPP technology is being investigated for the deposition of protective coatings for high temperature wear and erosion applications. MPP (modulated pulse power) is a variation of HIPIMS (high power impulse magnetron sputtering) that overcomes the rate loss issue through modulation of the pulse shape, intensity, and duration. In MPP, the pulse shape and duration and plasma perturbations directly affect the degree of ionization of the plasma. The typical pulse duration is in the range of 1-2 msec. In this study, tantalum and chromium films with thickness in the range of 10-100 microns are deposited using modulated pulse power. The films are deposited under floatings potential on surfaces of ASTM A723 steel components with complex geometry. The applied voltage pulse shape to the magnetron generated a high power pulse discharge and directly affected the degree of ionization of the sputtered material. The thickness and structure of each material was controlled by varying the output voltage pulse shape of the MPP plasma generator. The film structure, residual stress, and orientation are being investigated and film adhesion to the substrate is being tested. The deposition process and results from analysis of the films will be presented.
Monday, May 11

11:10 a.m. HP-5 Pulsed Magnetron Sputtering of Metallic Films Using a Hot Target

J. Vlcek, B. Zustin, J. Rezek, K. Burcalova, and J. Tesar, University of West Bohemia, Plzen, Czech Republic

Pulsed magnetron sputtering of titanium films was performed using a directly water-cooled target and a hot target of 100mm diameter to investigate the effect of the target temperature on the sputtering process. The repetition frequency of the pulsed dc power supply was 10 kHz at a 20% duty cycle and an argon pressure of 0.5 Pa. Almost constant target temperatures during depositions with the hot target, being heated by the ion bombardment itself, were controlled by preset values of the average pulse current at a target power density in a pulse up to 340 Wcm⁻². The temperature fields of the target surfaces were measured using a FLIR ThermaCAM SC2000 thermovision system. It has been shown that an increase in the surface temperature (up to 1700°C) of the hot target resulted in a rise in the deposition rate (up to 1.9 times) at a decreasing average pulse voltage (up to 1.5 times) compared to the cooled target with the same average pulse target current density (up to 0.33 Acm⁻²). The effects of the secondary electron emission and thermoemission on the discharge characteristics, and of an enhanced sputtering, sublimation and evaporation on the deposition rate will be discussed.

Monday, May 11

11:30 a.m. HP-6 Magnetron Configuration to Enhance Deposition Rate in High Power Impulse Magnetron Sputtering

A.P. Ehiasarian and A. Vetushka, Sheffield Hallam University, Sheffield, United Kingdom

High power impulse magnetron sputtering (HIPIMS) discharges are known to produce a highly ionised metal deposition flux with ionisation degree of up to 70%. It has been shown that the geometry of magnetic fields near the substrate can strongly influence the spatial distribution of film thickness. This research shows that varying the magnetic field strength at the cathode can influence the deposition rate without affecting uniformity. The magnetron's magnetic field was changed in a way that the shape of magnetic field near the substrates remained constant while the size of magnetic trapping tunnel near the target and the maximum tangential component Bt reduced. By decreasing Bt from 50 mT to 17 mT, the specific deposition rate of Cr increased from 270 to 360 nmh⁻¹kW⁻¹. Since the peak and average discharge power were maintained constant, the total peak plasma density remained constant at 9x10¹¹cm⁻³ as measured by Langmuir probes. The metal ion-to-gas ion ratio was high at 1.5:1 as measured with energy-resolved mass spectrometry. The ratio was maintained high for magnetic fields down to Bt = 25 mT and was associated with a dense film microstructure. As the field reduced to very low values of Bt = 17 mT, the metal ion-to-gas ion ratio dropped to 1:1 and the film microstructure developed well defined columns.
An energy/mass analyzer was used to characterize the plasmas during modulated pulse power (MPP) and conventional DC sputter deposition of Cr and reactive CrN films in a two-cathode closed field unbalanced magnetron sputtering system. Experiments were run with the different types of power applied to either just one of the cathodes or to both of the cathodes. The mass analysis detected Cr plus one, Ar plus one, and Cr plus two ions. The intensity of the Cr plus one ions when the MPP power is used is significantly higher compared to when DC power is used. As the peak power and the average power of the MPP pulse were increased, the intensity of the ions also increased in the closed field condition as it did when DC power was used. The energy analysis revealed that the average energy for the Cr and Ar plus one ions is about 2 eV and that the energy distribution is very small. There is a slight high energy tail to the ion energy distribution, but there is almost a mono-energetic source of ions from the MPP sputtering process.
Emerging Technologies
Monday, May 11

9:30 a.m. E-1 Photocatalytic Thin Films for Biomedical Applications

P.M. Martin, Columbia Basin Thin Film Solutions LLC, Kennewick, WA; W.D. Bennett, Pacific Northwest National Laboratory, Richland, WA; B.F. Monzyk, Battelle Memorial Institute, Columbus, OH; K.A. Dasse, Levitronix, Waltham, MA; and R.J. Gilbert, MIT, Boston, MA

Invited 40 min. Talk

Applications for thin films in biomedical applications are rapidly increasing. The photolytic artificial lung device being developed by Battelle Memorial Institute and Levitronix employs thin film photocatalytic materials, transparent conductive coatings, optical coatings and thin film membranes in a micromachined capillary structure. Progress toward the development of a photolytic artificial lung will be presented. The photolytic artificial lung is being developed to generate oxygen from water in blood using an ultraviolet light source in the presence of a catalytic surface. The basic structure consists of blood flow capillaries lined with a UV waveguide with an antireflection coating on the incident side to maximize photon transmission and quantum efficiency, a transparent conductive electrode, a microporous photocatalytic anatase titanium dioxide (2 - 5 mm thick), and a magnesium oxide coating in contact with the blood, all deposited by reactive magnetron sputtering. Blood flowed over the coated side and oxygen exchange occurred at the magnesium oxide interface. 0.354 nm UV radiation was incident on the silica/indium tin oxide side. Electron-hole pairs were generated in the titanium layer by the laser radiation, which catalyzed a redox reaction with water in the blood. The MnO₂ was also used as a catalyst to dissolve oxygen in the blood. This unique approach could obviate the need for exogenous gas, possibly reduce the blood-contact surface area and allow longer periods of patient support.

Monday, May 11

10:10 a.m. E-2 Comparison of Ion Trap and Quadrupole Sensors for Mass Spectrometry

G.A. Brucker, Brooks Automation, Inc., Longmont, CO

High vacuum partial pressure measurements using a novel ion trap based mass spectrometer sensor will be demonstrated. The details of the ion trap sensor, physical construction, excitation requirements and performance characteristics will be discussed, and compared to traditional quadrupole based mass filter sensor technology. Typical ion trap data will be presented for key performance metrics and used to illustrate the potential of this novel technology. Based upon a comparative analysis against quadrupole mass spectrometers, typical applications for the ion trap sensor will be described.
10:30 a.m. E-3 Effect of Growth Conditions on the Structure Stability, Transport and Magnetic Properties of Co Doped TiO$_2$ Films

B. Ali, University of Delaware, Newark, DE; A.K. Rumaiz, Brookhaven National Laboratory, Upton, NY; and A. Ozbay, E.R. Nowak, and S.I. Shah, University of Delaware, Newark, DE

Dilute magnetic semiconductors (DMS) are considered potential materials for spintronics devices. Although there is a controversy on the origin of the magnetism in DMS, there is some consensus on the role of oxygen vacancies in deriving the ferromagnetism (FM). In the present work pulsed laser deposited (PLD) thin films of cobalt doped TiO$_2$ on silicon and quartz substrates are investigated. Various oxygen partial pressures (PO$_2$) ranging from 6.6 mPa to 53 Pa are used during the deposition for the purpose of controlling the oxygen content in the samples. Crystal structure and transport/ magnetic properties of CoxTi$_{1-x}$O$_2$ ($x=0.01, x=0.03, x=0.06$) thin films are found to have a strong dependence on oxygen stoichiometry. X-ray diffraction (XRD) data revealed the presence of mixed phase material containing both anatase and rutile. However, the stability of each phase depends on the PO$_2$ present in the chamber during the growth of the films. The enhancement in electrical conductivity and magnetization is attributed to the off stoichiometric oxygen (oxygen vacancies). The activation energies obtained from the resistivity data fit to a simple thermal activation model are 20 to 140 meV. The experimental values of the activation energies are in a good agreement with the calculated values (~167 meV) for the binding energy of an electron bound to a defect site. Bound magnetic polaron model is adopted to explain the observed magnetic behavior of the samples.

Monday, May 11

10:50 a.m. E-6 Microwave Plasma-Assisted Chemical Vapor Deposition Homoeptaxial Synthesis of Single Crystalline Diamond

T. Schuelke, M. Yaran, D. King, and M. Becker, Fraunhofer USA, East Lansing, MI; and J. Asmussen, T. Grotjohn, and D. Reinhard, Michigan State University, East Lansing, MI

The mechanical, optical, chemical, thermal and electronic properties of diamond materials make it a desirable candidate for many applications, which includes, for example, high power and high frequency electronics. For electronic applications relevant diamond properties such as the band gap, saturated electron drift velocity, electric breakdown field strength and thermal conductivity exceed those of semiconductors such as silicon, silicon carbide, gallium nitride and gallium arsenide. Recent progress in the area of homoepitaxial synthesis of single crystalline diamond demonstrated material synthesis rates exceeding 50 microns per hour. This level of deposition rate may prove enabling for various diamond applications from a cost standpoint. The paper presents current results on the high rate homoepitaxial synthesis of single crystalline diamond including the simultaneous synthesis of 70 crystals.
Monday, May 11

11:10 a.m. E-5 Highly Insulating Al₂O₃, SiO₂ and Si₃N₄ Films for Sensor Applications Deposited by Reactive Pulse Magnetron Sputtering

P. Frach, H. Bartzsch, and D. Gloess, Fraunhofer Institute for Electron Beam and Plasma Technology FEP, Dresden, Germany; M. Gittner, Technische Universität IFE, Dresden, Germany; E. Schultheiss, Technische Universität IFE, Dresden, Germany and Fraunhofer FEP, Dresden, Germany; W. Brode, Siegert TFT GmbH, Hermsdorf, Germany; and J. Hartung, VON ARDENNE Anlagentechnik GmbH, Dresden, Germany

Applications in sensor, automotive and aviation technology require thin films that exhibit electrical insulating properties at room temperature but also at elevated temperatures. One technology for the deposition of such films is reactive pulse magnetron sputtering. Because of the high deposition rate this technology is especially interesting for the deposition of thick insulating films of several microns allowing high insulation voltages up to 800V or deposition onto relatively rough substrates e.g. stainless steel. In this paper the breakdown field strength and resistivity of such sputter deposited Al₂O₃, SiO₂ and Si₃N₄ films are investigated in the temperature range between room temperature and 400°C. All investigated films show excellent insulation properties at room temperature. At high temperatures, films remain insulating but leakage currents are increasing. The level of leakage currents is higher for Al₂O₃ than for SiO₂. The combination of different film materials allows fulfilling the requirements not only on insulating but also on thermo mechanical properties. Langmuir probe, stress and XRD measurements are used to discuss the results. One example of industrial application is the deposition of electrical insulation films onto the membranes of pressure sensors using cluster type sputter equipment.
Joint Session on Atmospheric Plasma Technologies
Monday, May 11

9:30 a.m. JAPT-1 Development of Adhesive-Free Lamination Technique Using a Plasma Surface Treatment at Atmospheric Pressure

M. Kogoma, Sophia University, Tokyo, Japan; A. Manabe, Fujimori Kogyo Co. Ltd., Yokohama, Japan; and K. Tanaka, Sophia University, Tokyo, Japan

*Invited 40 min. Talk*

The laminated polymer films are widely used for the packaging materials of foods, cosmetics and so on. These laminated films are generally two or more kinds of polymer films pasted together with an organic adhesive diluted by the toxic organic solvent. Thus, a new adhesive-free lamination technique is desired. In this study, we tried to develop an adhesive-free lamination technique using the atmospheric pressure glow (APG) plasma surface treatment and the thermo-compression of the treated super posed films. The popular lamination materials, PET/PE, NY/PE were treated by N$_2$ /He or He APG plasma reactor. The reactor can treat the film surfaces faster than 10 ms/cm. The adhesive strength of the laminated film was measured with a 180 degree peel tester. In the results, the maximum adhesive strength of both materials were attained much higher value (>500 N/m) than that of the required in the industry. XPS spectra in the valence band region of the treated LDPE surfaces show some decreasing of the main peak obtained around 15 eV that should be related to C-C bonds of the PE structure. We supposed that some low molecular weight PE were produced on the film surface and that they acted as an adhesive for the heat-press lamination.

Monday, May 11

10:10 a.m. JAPT-2 A Novel Atmospheric Microplasma Source with Integrated GaN HEMT Microwave Power Oscillator

R. Gesche, S. Kuehn, and H. E. Porteanu, Ferdinand-Braun-Institut, Berlin, Germany; and R. Kovacs and J. Scherer, Aurion Anlagentechnik GmbH, Hessen, Germany

A novel atmospheric microplasma source is presented where the microwave power is generated by an integrated power oscillator, which is based on a GaN HEMT transistor. A microwave resonator acts as plasma electrode, performs the impedance transformation for ignition and plasma operation and determines the oscillation frequency. The source operates at a frequency of around 2.45 GHz with a maximum oscillator power of 30 W. The size of the complete source module including electrode, resonator and microwave oscillator is as small as 30 mm by 30 mm by 40 mm, the visible afterglow plasma flame has a diameter of approx. 1 mm and a length up to 5 mm. The paper describes the concept and presents procedure and results of ignition voltage and impedance measurements. Ignition voltage of air at atmospheric pressure is found to be less than 400 V. As a first application example, we present data on surface energy enhancement. Contact angle profiles on several polymers we show shown as a function of the distance between source and polymer. Promising activation results are achieved for oxygen as well as for air (nitrogen) plasmas.
10:30 a.m. JAPT-3 Influence of Substrate to Source Distance on the Properties of Siloxane Coatings Deposited Using an Atmospheric Plasma Jet System

D.P. Dowling and M. Ardhaoui, University College Dublin, Dublin, Ireland

Atmospheric plasmas combined with liquid deposition of precursors can be used to deposit a wide range of functional coatings onto ceramic, metal and polymer substrates. In this study a TEOS precursor is nebulised into a helium plasmas formed using a PlasmaStream system. In this system the atmospheric plasma is formed between two metallic electrodes and has an operating frequency in the range 15-25 kHz. A Teflon tube is mounted at the orifice of the applicator with length of 75 mm and diameter of 15 mm. Due to the flow of He gas, the plasma extends approximately 10 mm out from the base of this tube. The size of the plasma flame is dependent on the He flow rate and in this study it was systematically altered between 2 and 30 l/min. The silicon wafer to jet orifice distance was also changed from 1 to 10 mm and the effect of changing both of these parameters was investigated on deposited siloxane coating properties. Due to increased air mixing siloxane coatings deposited at larger substrate to nozzle distances and lower He flow rates exhibited more SiOx rich chemistry. This was demonstrated based on XPS analysis, FTIR spectroscopy and contact angle measurements. The area of coating coverage was measured using spectroscopic ellipsometry; coating thickness was mapped across the wafer. As anticipated with increase distance from the wafer the area of coating coverage on the 6 cm diameter wafers increased, but the maximum coating thickness directly under the jet decreased.

10:50 a.m. JAPT-4 Biomedical Applications of Atmospheric Pressure Plasma

K.-D. Weltmann, Th. von Woedtke, R. Brandenburg, and J. Ehlbeck, INP Greifswald e.V., Greifswald, Germany

Progress in life sciences is increasingly caused by utilization of unrelated technologies and knowledge. Microelectronics, optics, or material sciences as well as nanotechnology, nowadays, are key technologies in modern medicine. A similar trend can be expected concerning plasma technology. Plasma medicine can be subdivided into three main components: plasma surface modification, plasma bio-decontamination, and, as the central field, therapeutic plasma application. The scientific basis of plasma medicine is a fundamental knowledge of the mechanisms of plasma interaction with living cells and tissue. In the areas of plasma surface modification and plasma bio-decontamination, plasma is used to treat surfaces and products to improve their bio-applicability or bio-performance to use it for therapeutic purposes. Whereas in these fields, both low-pressure and atmospheric pressure plasmas can be used, for direct therapeutic plasma applications only atmospheric pressure plasma sources can be used. An extremely promising field will be the plasma-based treatment of chronic wounds. A selective antimicrobial (antiseptic) activity without damaging surrounding tissue, combined with a controlled stimulation of tissue regeneration could revolutionize wound care. Other fields are the treatment of skin diseases, tissue engineering, or tumor treatment based on specific induction of apoptotic processes. Selected plasma sources will be discussed regarding possible applications in this field. Results of the treatment of cells and clinical relevant microorganisms will be presented.
The recent introduction of high density atmospheric plasmas for treatment of powders, thin films, fibers and woven fabrics has overcome the large cost of the conventional low pressure (vacuum) plasma systems. The main limitations of the latter are limited throughputs and the higher capital and operation costs of vacuum equipment. Atmospheric pressure plasmas, however, offer the potential for high throughput, roll-to-roll wide web treatments due to the high species flux, inherent to atmospheric pressure operation. In this work, we present results from the surface modification of various polymer films and fibers due to the exposure to a helium-oxygen, nitrogen, and air dielectric barrier discharge (DBD), operating under atmospheric pressure. Contact angle, X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM) data, and atomic force microscopy (AFM) are presented and the dependence of the surface chemical and topological changes on the discharge parameters such as treatment time and gas composition is investigated. Experimental results reveal improved hydrophilicity of the plasma exposed polymers and an increase of their surface energy. This can be attributed to the surface functionalization during plasma treatment, as confirmed by XPS analysis. SEM and AFM data show changes in the surface morphology and roughness depending on the plasma processing conditions, suggesting that mild etching occurs in a controlled fashion. The above observations indicate that the plasma treatment leads to an improvement of the surface properties such as wettability and adhesion and can be therefore used in a variety of applications. Standard lap-shear evaluations reveal that plasma treatments lead to significant increases in the bond strength of polymer films and resins. Most importantly, this uniform modification occurs within a few seconds of exposure, time comparable to continuous on-line industrial processing.

Smooth, homogeneous and transparent SiO2 layers deposited on wide area polymers are receiving much attention in packaging industries as well as in the field of encapsulation of flexible electronics. This layer deposited in AP-PECVD, using dielectric barrier discharge, is considered as a promising technology due to its economical and ecological advantages. The control on the layer properties requires a deep understanding of the plasma physics and film growth mechanisms. This work reports the predominant growth processes involved in AP-PECVD and their contribution to the quality of SiO2 over polymeric substrates fed with Ar-N2-O2-hexamethyldisiloxane (HMDSO) mixtures in a roll-to-roll configuration. Detailed surface analysis on films (thickness, composition, morphology, roughness and homogeneity) grown under static and web roll conditions with various gas flow directionalities unraveled the different stages of growth involved in the deposition of smooth and uniform layers, as being controlled by the diffusive flux of HMDSO radicals generated in the plasma as well as by the surface modification of the polymer substrate by the reactive species (O, OH). The negligible influence of morphology and roughness with film thickness (up to 300 nm) and comparable to that of uncoated polymer indicates the SiO2 growth follows the topology of the substrate.
Vacuum Web Coating
Monday, May 11

9:30 a.m. W-1 Polyester Substrates and Vacuum Deposition: Process Optimisation for Improved Product Performance

M. Hodgson, Dupont Teijin Films UK Ltd., Wilton Centre, United Kingdom

Invited 40 min. Talk

Sputter deposition of various materials on PET webs is influenced by the low pressure environment during coating and by particular aspects of the PET surface itself. Optimising this coating/substrate interaction is of primary importance when considering the applications into which sputter coated PET substrates will be used (e.g. flexible electronics, optical films etc.), and it is essential that this interaction can be probed. In order to achieve this, characterisation of the surface before and after coating has been used to yield important information with respect to the deposition process. However, this characterisation itself is fraught with difficulty due to the anisotropic nature of biaxially drawn PET film. In this regard, careful and precise measurement of optical properties is critical to ascertain how sputter deposited layers, particularly metal oxides, interact with polyester surfaces. Ellipsometry, microscopy and optical spectroscopy have been used to ascertain the surface changes which occur during deposition and this knowledge is used to optimise the process and improve product performance.

Monday, May 11

10:10 a.m. W-2 Transparent Conducting Oxides on Polymer Substrates

S. Louch, Centre for Process Innovation, Redcar, United Kingdom; and M. Hodgson, Dupont Teijin Films, Middlesbrough, United Kingdom

This presentation will look at the sputter deposition of two important transparent conducting oxide materials; Al doped ZnO and ITO, onto plastic substrates using a roll-to-roll coater. Deposition from both metallic and ceramic targets is considered and differences in coating efficacy highlighted. A variety of techniques used to characterise these coatings will be described, and important factors such as the limitations of the substrate type and the deposition parameters will be discussed. Finally, the properties of these types of coatings specific to flexible electronic applications will be considered in view of the move towards high volume production of flexible electronic products.
Monday, May 11  
10:30 a.m. W-3 Optical, Electrical, and Structural Properties of ZAO and ZGO Coatings Deposited by Magnetron Sputtering onto Plastic Substrate

R. Kleinhempel, R. Thielsch, and A. Wahl, Southwall Europe GmbH, Grossroehrsdorf, Germany

Transparent conductive oxides (TCO) with various electrical properties are essential functional parts in display applications (touch panels and LCD) or OLEDs. Tin doped indium oxide (ITO) still provides the best electrical properties at high optical transparency. Due to raising indium prices alternative TCO materials like aluminum or gallium doped zinc oxide (ZAO resp. ZGO) are the subject of intense work. The goal is to understand influences of deposition conditions in order to improve the functional properties. While deposition onto glass at elevated temperature gives best results so far, challenging demand for improved and optimized properties of doped zinc oxide thin films arise from the general trend of light weight, large area as well as from miniaturized display applications that force the use of organic substrates. The paper reports about sputtering a zinc oxide target with different dopants (Al, Ga) at different sections of the target in a production size machine onto PET. This enables a direct comparison of functional properties of ZAO and ZGO films deposited at equal process conditions as well as analysing of Al / Ga co-doped films with various concentrations. Electrical, optical and structural properties are discussed in dependence on deposition conditions and compared to ITO properties.

Monday, May 11  
10:50 a.m. W-6 Low Temperature Deposition of AZO Coatings on Polymeric Web

P. Barker, G.T. West, and P.J. Kelly, Manchester Metropolitan University, Manchester, United Kingdom; and J.W. Bradley, University of Liverpool, Liverpool, United Kingdom

It would be significant to industry if TCO coatings, such as aluminium-doped zinc oxide (AZO) could be readily deposited onto flexible polymeric web, rather than onto rigid glass substrates. This would provide reductions in weight and cost of the finished products, whilst also increasing throughput and efficiency by utilising roll-to-roll web coating technology. The thermally sensitive nature of the substrates, though, currently limits the choice of deposition process. However, HiPIMS (high power impulse magnetron sputtering) may provide a solution to this problem. Despite the very high peak powers (up to MWs) achievable in this mode, the thermal energy flux to the substrate has been shown to be significantly lower, compared to other magnetron sputtering processes. Furthermore, the process also produces high levels of ionisation of the target material, which offers the potential to produce high quality TCO coatings on polymeric web without the need for post annealing processing. This paper discusses the deposition of AZO coatings onto PET web through the use of HiPIMS. The coatings have been characterised in terms of their structural, optical and electrical properties.
11:10 a.m. W-7 Deposition of High Mobility ZnO and InZnO Thin Films at Ambient Temperature Using HiTUS Based Technology for TFT Applications

J.D. Dutson, Plasma Quest Ltd., Hook, United Kingdom; A.J. Flewitt and P. Beecher, University of Cambridge, Cambridge, United Kingdom; and S.J. Wakeham and M.J. Thwaites, Plasma Quest Ltd., Hook, United Kingdom

InZnO for TFT applications. Using the novel HiTUS deposition system to remotely generate a plasma for the deposition process, it is possible to decouple the ion energy and ion density providing greater control of the deposition variables and an increased densification of the final film. This technique has been used to sputter deposit amorphous ZnO and InZnO films using a reactive deposition process with no substrate heating. The control provided by the HiTUS system enables the conductivity of the layers to be varied by 13 orders of magnitude (from $10^{-9}$ to $10^{+4}$ $\Omega$^{-1} m^{-1}) by varying the oxygen flow rate alone. Hence, both semi-conductor and charge injection layers can be deposited by the same process. These layers have been used to produce TFT devices on a variety of substrates and dielectric insulator films. The switching properties of the complete devices have been measured and a switching ratio of $>10^6$ and field effect mobility for ZnO\(\approx\)0.2 cm\(^2\)V\(^{-1}\)s\(^{-1}\) and InZnO\(\approx\)10 cm\(^2\)V\(^{-1}\)s\(^{-1}\) have been extracted. In the full paper we describe the deposition criteria in full and present the complete set of results for the TFT devices.

Monday, May 11

11:30 a.m. WFT-2 Paucity of Materials, a Potential Constraint to the Growth of Many Markets Including Displays and Photovoltaics

C. Bishop, C.A. Bishop Consulting Ltd., Loughborough, United Kingdom (Presented by G. Tullo, General Vacuum Equipment, Raleigh, NC)

There has been plenty of publicity about the rapid increase in the price of indium. This price increase was the result of factors, such as it only being mined as a by-product of zinc and stockpile limitations. This was exacerbated by a large increase in its use for transparent conducting coatings for the display industry as well as the newer and faster increase of use within the photovoltaic industry. The net result of this was that companies suddenly found that recycling made sense and so the supply was enhanced, at least for a time, by the input of recovered indium. Observation of the metals trading markets indicate all kinds of price increases in materials that heretofore were considered abundant. In reality with the increased world population and increasing affluence, the demand for goods is increasing and the amount of materials being tied up in these goods is increasing. This requires mining more materials and, for some, this is becoming more difficult and expensive as these materials are found to be increasingly scarce. A number of these materials have already passed their peak in world production and yet, in some markets, predictions of market growth appear to be oblivious to the dwindling resources that will cause inevitable shortages. These shortages are likely to affect a whole range of products from fluorescent lights, photovoltaics and light emitting diodes (LEDs). All of these are products that are aimed at reducing energy consumption of for energy production. In this paper some of the materials that are predicted to be under threat are highlighted.
Monday Afternoon
May 11, 2009
Advances and Drawbacks of Microwave Plasmas

M. Moisan, Groupe de Physique des Plasmas, Université de Montréal, Montréal, Canada

Invited 40 min. Talk

As compared to conventional RF capacitive or inductive discharges, microwave-sustained plasmas exhibit a few specific characteristics that make them original in terms of physics and technology: i) the electric field sustaining the discharge is provided by wave propagation either along dielectric materials (including the plasma itself) or within the structure of field applicators that radiate outwardly to penetrate dielectric discharge vessels (transparent to microwaves): no electrodes need to be in contact with the discharge and, therefore, no self-biasing of the field applicators; ii) in the low pressure range there exists a highly efficient power absorption mode, the electron cyclotron resonance (ECR); iii) impedance matching is easier and more efficient than with RF systems. Despite the attractiveness of their features, microwave plasmas are suffering from severe difficulties when compared to other technologies (corona and dielectric barrier discharges at atmospheric pressure, RF discharges at reduced pressure) in the two main sectors of industrial applications, namely chemistry in gaseous phase and plasma processing of surfaces. Drawbacks are essentially: i) the (radial) contraction of microwave (tubular) discharges typically at pressures above 10-20 Torr and, additionally, their filamentation at frequencies above 1 GHz; ii) at reduced pressures (< 500 mTorr), the difficulty of scaling up microwave plasma sources for surface treatments. The scaling-up solutions adopted some twenty years ago with the distributed ECR (DECR) and duo plasma-line schemes calling on the distribution of the sustaining microwave-power field through an array of parallel linear sources still present strong limitations, basically due to the difficulty of achieving uniform standing wave patterns over the full length of long applicators. Recent advances in microwave plasma sources are addressing the above problems. At and above atmospheric pressure, we have shown that operation of tubular discharges at power density up to 1.4 kW/cm³ at 6 bars is possible thanks to the design of efficient cooling systems. At reduced pressure, plasma scaling-up without limitation is now a reality thanks to new concepts based on wave propagation on external-to-the plasma non-dissipative structures or on the distribution of elementary plasma sources on two-dimensional (planar sources) or tri-dimensional networks (plasma volume) that include the so-called multi-dipolar and matrix plasmas. Impedance matching is definitely more efficient and reproducible with microwave components than RF match-boxes: as an example, we consider the case of surface-wave discharges sustained with the optimised surfaguide applicator that yields an impedance matching almost independent of operating conditions. All of these new features represent important breakthroughs. Design, performance and applications of these new generations of microwave plasma sources are reviewed.
Vacuum Processes and Coatings for Healthcare Applications
The Effect of Surface Topography on the Retention of Organic Soil and Microorganisms

A. Packer, P.J. Kelly, K.A. Whitehead, J. Verran, and G.T. West, Department of Biological Sciences, Manchester Metropolitan University, Manchester, United Kingdom

Wear of food contact surfaces through abrasion, cleaning and impact damage increases the surface roughness and introduces topographical features which may increase the retention of organic soil and microorganisms. This affects the hygienic status of the surface by providing protection for the microorganisms and interfering with disinfection protocols. To investigate this, surfaces with defined linear features were fabricated and coated with titanium to provide a uniform surface chemistry. Retention assays were performed using Staphylococcus sciuri and Escherichia coli. Using atomic force microscopy (AFM), it was shown that interactions between cell shape and surface feature size affected the force required to remove cells. In order to model realistic situations the Crock Meter was used to assess the microbial removal in the presence of an organic soil. The results showed differing levels of retention dependent on the topography of the surface and the presence of an organic food soil. For example, E. coli was retained poorly on titanium coated surfaces without the organic soil.

Hydrogen-Free Tetrahedral Amorphous Carbon Coatings for Biomedical Implants

L. Haubold, M. Becker, T. Schuelke, and H.-J. Scheibe, Fraunhofer USA, East Lansing, MI; and G. Woodrough and J. Helmuth, Symmetry Medical Jet, Lansing, MI

Hydrogen-free tetrahedral amorphous carbon (ta-C) coatings represent the hardest and most wear resistant form of diamond-like carbon films. In addition to their exceptional wear performance these coatings provide corrosion resistance and biocompatibility, which makes them a desirable candidate for biomedical implant applications such as hip and knee joint replacements. A very efficient laser-controlled high pulse-current cathodic arc technology was used to deposit thick (up to 10 microns) ta-C coatings onto actual implant components made from cobalt-chromium steel. The components were tested in a commercial hip-joint simulator for corrosion and wear performance. One of the key factors that affect wear performance in biomedical implant applications is the surface roughness of the coated implant part. The paper presents process information and data on how the wear performance of the ta-C coating depends on the surface roughness.
Monday, May 11

2:10 p.m. HC-3 Sterilisation of Surgical Instruments Using Mini Electron Accelerators

G. Gotzmann and F.H. Roegner, Fraunhofer Institute for Electron Beam and Plasma Technology FEP, Dresden, Germany

Invited 40 min. Talk

Electron beam technology has been used for sterilisation applications for several decades. The process is accepted all over the world and complies with international standards (ISO 111-37/95). The aim of the development work at the Fraunhofer Institute for Electron Beam and Plasma Technology (FEP) is to develop in-line-capable systems that can be used both for sterilisation applications in production processes and also as a batch system. The rapid growth of the older population, as well as modern medical technology, requires the increased use of gentle and minimally invasive diagnostic methods and devices. The delicate construction of these devices makes the sterilisation more difficult. This is one of many reasons for more research into alternative conditioning processes for medical devices. The fitness for use of accelerated electrons for this range of sterilisation problems was studied at the FEP. To assess the success of sterilisation, approved microbiological methods were applied. Further energetic and morphological changes in the treated surfaces were estimated. The results of electron beam sterilisation were compared with those of traditional sterilisation methods. The results show that electron beam technology can become an alternative method for sterilization, e.g. of surgical instruments and other medical products. Further investigations are planned.

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Monday, May 11

2:50 p.m. HC-4 Validation Concepts for a Biomedical Product

P.E. Gagnon and J. Gibbs, Corning Inc., Kennebunk, ME

Developing and manufacturing bio-medical products require a battery of upfront testing. As pharmaceutical companies develop more complex drugs, they rely more and more on product validation from manufacturers. This paper describes some of the validation testing needed to meet bio-medical requirements. These include: bioburden, biocompatibility, shelf life, class IV testing, pyrogen and discussions of USP testing.
Gas jet deposition of thin films and nanoparticles has gained wide development in the last decades for different technical applications. The case in point is the synthesis of condensed deposits in the result of interaction of rarefied subsonic or supersonic jets with a target. The deposition can be run in free molecular, transition or continuum regimes. This paper presents the review of current elaboration of new gas jet deposition methods, the results of experimental investigations of polymer and metal-polymer film deposition, informative illustration on computational simulation of non-equilibrium processes in the gas-precursor flow and data on effect of silver-polymer coating, inhibiting some dangerous bacteria. The numerical simulation of interaction of supersonic flows with a substrate provides data on distribution of macroscopic thermodynamic parameters in the flow and characteristic of internal energy of molecules, where it is necessary. The study of synthesis of polymer films is oriented on deposition of teflon-like films from supersonic jets of C_2F_4 substrates with given temperatures. New possibilities are open with the use of the hot wire initiation of supersonic precursor flow. The gas jet deposition was used for synthesis of metal-polymer film by co-deposition from separated jets of C_2F_4 and mixture of silver vapor and argon. This coating destroys the Salmonella typhus completely.
Tribological and Decorative Coating
Monday, May 11

1:30 p.m. T-1 Novel Design Approaches to the Development of Multifunctional Nanocomposite Coatings for Demanding Engine Applications

A. Erdemir and O.L. Eryilmaz, Argonne National Laboratory, Argonne, IL; and M. Urgen, K. Kazmanli, and V. Ezirmik, Istanbul Technical University, Istanbul, Turkey

*Invited 40 min. Talk*

In this paper, we describe a crystal chemical model that can help identify the kinds of coating ingredients that are needed in nano-composite coatings for achieving ultra-low friction and wear under boundary lubricated sliding conditions. Using this model, we recently designed and synthesized a series of MoN-based nano-composite coatings and confirmed their superior tribological properties under severe sliding conditions. Employing advanced analytical tools (such as time-of-flight secondary ions mass spectrometry, x-ray photoelectron spectroscopy, and Raman spectroscopy) we ascertained the chemical nature of the tribofilms forming on sliding surfaces of these MoN-based nano-composite films that were responsible for their superior friction and wear properties. Overall, crystal chemical model used in this study seems to provide a new scientific means for the design and production of next generation nanocomposite coatings that can endure harsh tribological conditions of various engine applications.

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Monday, May 11

2:10 p.m. T-5 Effect of Si Incorporation on the Mechanical and Electrochemical Properties of DLC Films

M. Azzi, École Polytechnique de Montréal, Montréal, Canada and McGill University, Montréal, Canada; M. Paquette, École Polytechnique de Montréal, Montréal, Canada; J.A. Szpunar, McGill University, Montréal, Canada; and J.E. Klemberg-Sapieha and L. Martinu, École Polytechnique de Montréal, Montréal, Canada

In the present work, the effect of silicon incorporation on the mechanical, tribological and electrochemical properties of diamond like carbon (DLC) coatings on metal substrates is investigated in the context of their biomedical applications, namely in implants or in surgical instrumentation. DLC films with different silicon contents (0, 16, 20, 26 at.%) were deposited using plasma enhanced chemical vapour deposition (PE-CVD). Elastic recoil detection (ERD) technique was used to measure the chemical composition, and nano-indentation tests were carried out to assess the mechanical properties of the DLC/Si films. The hardness slightly deceased with increasing the Si content from about 18 GPa at 0 at.% Si to 16 GPa at 26 at.% of Si. Electrochemical tests were performed in Ringer’s solution to simulate the body fluid conditions. Potentiodynamic polarization curves were measured and corrosion current densities, passive current densities, and breakdown potentials were obtained. Electrochemical impedance spectroscopy (EIS) was applied to measure the resistance to general corrosion (RGC) of the coatings. EIS spectra were interpreted in terms of appropriate equivalent electrical circuits. It was found that RGC increased significantly with increasing Si content from 3.5 GΩ.cm² at 0 at.% to almost 25 GΩ.cm² at 26 at.%. This finding represents a clear indication of lower porosity of the DLC that can be related to enhanced oxidation of Si inside the film. Potentiodynamic polarization curves showed that the passive current and the breakdown potential increased with Si incorporation; in fact, the passive current decreased almost one order of magnitude over the studied range (0 at.% to 26 at.%). The Si-doped DLC coatings, in conjunction with appropriate interface engineering approaches on metals, and the use of highly resistant interfacial layers provide excellent protection against tribo-corrosion effects in liquid (particularly body fluid) environments.
Monday, May 11

2:30 p.m. T-3 Reactive Co-Evaporation of Carbon/Carbide Nanocomposites: Process, Structure and Tribological Properties

E. Bergmann, University of Applied Science of Western Switzerland, Geneva, Switzerland; G. Wahli, Roth & Rau AG, Neuchâtel, Switzerland; G. Pannatier, Platit AG, Grenchen, Switzerland; B. Pecz and L. Toth, Muszaki Fizikai és Anyagtudományi Kutatóintézet, Budapest, Hungary; and C. Mitterer, University of Leoben, Leoben, Austria

Reactive co-evaporation is a new method to synthesize carbide/carbon nanocomposite coatings. Coatings are produced by cathodic arc evaporation from solid metal and carbon sources. The method can be tuned to produce almost isotropic nanocomposites. They are hydrogen and metal free. The coatings are thermally stable to at least 900˚K. Their hardness is in the range of 13-16 GPa, slightly lower than CrN. The structure of CrC/C and CrCN/C coatings will be presented. Depending on their composition, the coatings have low friction at room temperature, but maintain fair tribological properties up to higher temperature.

Monday, May 11

2:50 p.m. T-4 Erosion-Resistant Multilayer Coatings

A. Flores Renteria, O. Schroeter, R. Mykhaylonka, and C. Leyens, Technical University of Brandenburg at Cottbus, Cottbus, Germany

In this research work, the development of an alternative erosion-protective coating for compressor blades has been carried out. In those parts of the world where the sand particle concentration in the air is high, the lifetime of the compressor blades is principally limited by erosion. The sand particles are ingested into the aero-engine during low-altitude flights and upon take-off and landing. The use of coatings is one standard method to effectively protect bulk materials against erosion. Particular attention has been given to multilayer coatings containing hard and tough-ductile layers. In this study, the Cr₃C₂/Cr₂AlC multilayer system was investigated. The Cr₃C₂ layers are hard enough to avoid particle penetration, and the Cr₂AlC phase should be able to resist the deformation and cutting processes caused by the impacting particles. The coatings were deposited by DC-magnetron sputtering, and the erosion tests using solid particles were carried out in an erosion rig. The obtained results indicate the effective protection of metal alloys against erosion of solid particles impacting at different angles and velocities, which are representative of in-service conditions for compressor blades.
Plasma Processing
Monday, May 11

1:30 p.m. P-1 Novel Control of Surface Energy for Functional Metallization by Integrated Diagnostics of Processing Plasma

J.G. Han, N. Britun, Y.J. Kim, and Y.S. Choi, Sungkyunkwan University, Suwon, South Korea

Invited 40 min. Talk

It is noted that thin film synthesis by plasma processing is hard to be tailored as designated microstructure and corresponding properties due to inherent complex parameters to control and monitor. Therefore it is crucial to understand the theoretical mechanism of film nucleation and growth for precise control of microstructure and corresponding film properties, and then design for designated film structure and synthesis process. The film nucleation and growth is closely controlled by the total energy to be delivered to the substrate surface by energetic particles and other external energy sources such as heating or cooling during plasma processes. This paper discusses on the mechanism of energy transfer at the substrate surface during magnetron sputtering and PECVD, and changes of particles energy and surface temperature with variation of process parameters during magnetron sputtering and PECVD by integrated plasma diagnostics and related energy simulation. The design of thin film structure and process is then discussed for synthesis of metal and oxide films with specified microstructure and properties and compared with empirical data measured for the films deposited by magnetron sputtering and PECVD at various process conditions. The design principle is finally proposed for functional metallization at low temperature by magnetron sputtering and PECVD.

Monday, May 11

2:10 p.m. P-2 Negative Ion Density Measurement by Photo-Detachment in RF and Pulsed DC Magnetron Discharges

S.D. You, R. Dodd, P.M. Bryant, and J.W. Bradley, University of Liverpool, Liverpool, United Kingdom

Sponsored Student Presentation

Reactive magnetron sputtering, in the presence of oxygen, is an important industrial technique for depositing engineering quality dielectric and ceramic thin films. Despite their success, there is still a lack of understanding of the role of negative ions in these discharges and their influence on the growth of the deposited films. Here we present detailed photo-detachment measurement of the negative ion densities (O⁻, O₂⁻) in both RF and pulsed DC driven magnetron devices. The photo-detachment technique uses an Nd: Yag laser at 1054 nm and 532 nm, in conjunction with a Langmuir probe data collection system. Preliminary measurements show negative ion densities are greater than the electron densities in low power RF sputtering. In the case of DC operation, the negative ion density N⁻ was found to be 3.6x10⁻¹²m⁻³ which is significantly lower than that obtained in the RF magnetron where N⁻ was about 1.7x10⁻¹⁴m⁻³ under similar conditions, namely at 10 mTorr and 40 W power. The technique will be developed further for high power RF and pulsed DC reactive sputtering in oxygen-argon mixtures, yielding time-averaged negative ion densities in the bulk plasma.
2:30 p.m. P-3 Electrical Probes for Monitoring Electron Density, Ion Flux and Film Properties in Deposition Plasmas

N.S.J. Braithwaite, Department of Physics and Astronomy, The Open University, Milton Keynes, United Kingdom

*Invited 40 min. Talk*

The densities and fluxes of various charged particles are key parameters affecting the rate of many plasma processes and thereby provide a means of process control. Monitoring of these quantities by means of electrical probes is especially challenging in deposition environments: conducting material tends to short out insulating parts of a probe structure; insulating deposits block the current path between the plasma and the external circuitry. There are nevertheless several electrical probe techniques that can be used to provide real-time monitoring signals even in the face of substantial deposition of material, whether conducting or insulating. For instance, a simple 1-2 cm long hairpin structure has been used to track the electron density in a magnetron source. This method uses microwave-resonances of the hairpin and provides sub-microsecond resolution. In the same system a transiently biased planar probe has been used to follow the flux of ions onto targets and substrates on sub-millisecond time scales. The probe can tolerate several microns of insulating deposit because the method measures only transient current. This scheme has also provided real-time assessments of the thickness of insulating coatings; in addition, subsequent analysis of the transient behaviour yields characteristic *in situ* dielectric properties of the coating.
Large Area Coating
Monday, May 11

1:30 p.m. L-15 Reliable Design, Installation, and Operation of Large Industrial Turbomolecular Pumping Systems as an Energy Reduction Strategy for Large Area Coaters

T.C. Forbes, Varian, Inc., Lexington, MA and E. Emelli, Varian, Inc., Leini, Italy

Turbomolecular pumps (turbo pumps) are now the main high vacuum pumping choice for designers of large area coating systems in order to reduce power consumption. This is because these pumps provide dry, high vacuum and high throughput performance in a compact, low power consumption package compared to traditional diffusion pumps. On the other hand, there are key issues to consider when designing a coater's vacuum system to insure effective and reliable operation. These include proper turbo pump and primary pump sizing, considerations on the nature of the pumped gases, cooling (utilities) requirements, and most importantly, the need to verify and monitor all these parameters throughout the operating life of the system. This paper describes the basic vacuum system design parameters needed to minimize power consumption, the application and installation requirements to ensure reliable and effective operation of these pumps, and methods of monitoring the vacuum system during operation.

Monday, May 11

1:50 p.m. L-11 Stay Green - Optical Thickness and Composition Control for Large Area Coaters

W. Theiss, W. Theiss Hard- and Software, Aachen, Germany

Thin film deposition in large area coating equipment has to be continuously monitored. This is most easily be done by optical spectroscopy. In case of deviations from the wanted coating properties, the optical measurements need some interpretation in order to make appropriate decisions about changes of the deposition settings. We show how optical modeling can provide the desired information about the deposited thin film systems. Thickness and composition changes of the layers are identified and feedback to the operators is given proposing new machine settings. This way the production can be run for a long time with the coating product staying within allowed tolerances. The required network of spectrometer hardware and software components is discussed, as well as problems that arise when optical modeling is added to existing simple monitoring systems.
Titanium dioxide is among the few semiconductors that have good chemical/photochemical stabilities and high oxidation power. However, its relatively high band gap makes it only effective when exposed under UV light. It has been found that the addition of transition metals to TiO₂ can improve the photocatalytic activity by UV irradiation and extend its use in the visible region of the electromagnetic spectrum. The photocatalytic properties of TiO₂ films deposited on polycarbonate have rarely been reported in physical vapour deposition (PVD) industry. In fact, polycarbonate is one of the newer thermoplastics used in the construction and automotive industry due to its excellent properties of impact resistance, low weight and transparency.

Transparent titanium dioxide (TiO₂) thin films were deposited onto microscope glass slides by means of the DC reactive magnetron sputtering method. The films were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), atomic force microscopy (AFM), UV-visible spectroscopy (UV) and contact angle analysis using the Owens-Wendt method for the surface energy calculation. The photocatalytic activity of the films was tested by measuring the photodegradation of Rhodamine-B (RhB) dye under radiation of UV light. Iron-doped TiO₂ films were also prepared in order to study the Fe-doping effect on TiO₂ photocatalytic activity. The influences of different iron concentrations on the contact angle of the series of Fe-doped TiO₂ thin films, were investigated. The influences of total sputtering pressures on TiO₂ photocatalytic activity were also investigated. It was observed that the photocatalytic activity of the TiO₂ thin films was slightly improved by increasing the total sputtering pressure. Moreover, it was also observed that in general, iron doping was detrimental for photocatalytic activity, nevertheless the films with low iron concentrations showed better photocatalytic activity than those with higher iron concentrations. It was found that iron doping has changed the wettability appetency of TiO₂ coated surfaces.

Rotary magnetrons offer many advantages, both in terms of coating quality improvements and in terms of production cost. Although the sputtering targets needed for such magnetrons are normally much more expensive than their planar counterparts, the total cost of ownership of rotary ceramic ITO-targets turns out to be considerably lower than for planar ceramic targets. This is due to higher process yield (less arcing, reduced particles and pinholes), higher target utilization and better collection efficiency, lower energy and labour costs, higher system up time. Unfortunately, many PV-manufacturers just take the target cost per kilogram into account when they make technological comparisons. This work will prove that a more in-depth cost analysis is needed using the example of a transparent conductive oxide (TCO-coating) as is used in TFT-LCD’s (colour filter ITO) or flexible thin film PV cells. In the latter application, this TCO is typically responsible for more than 5% of the front end production cost and its characteristics contribute considerably to the cell efficiency. Hence, it is worthwhile focusing on this component of the thin film cell.
Buildings consume up to 40% of the energy used in the US and the European Union. Of the energy losses from a building, windows are a primary source. To increase energy efficiency, architects and builders have turned to low-e windows. Switchable windows based on electrochromics, however, shows significantly better energy efficiency. When compared with an efficient low-e window with the same day-light control system, a switchable electrochromic window shows annual peak cooling load reductions from control of solar heat gains of 19-26% and lighting energy use savings of 48-67% [LBNL "PIER" study for California]. In addition to superior energy efficiency, electrochromic windows offer a number of other advantages including eliminating the need for blinds and shades, and blocking heat and glare while still keeping the view. Despite the clear benefits, several hurdles must still be overcome before electrochromic windows can enjoy widespread applications in the architectural market, as well as the automobile market. These hurdles include: 1) durability, especially in tough environmental conditions such as heat, cold, and sunlight's UV, 2)sufficiently large optical switching range with high uniformity and acceptable colors, 3) scalability to large sizes, and 4) cost. Much progress and advances have been made the last few years to address these hurdles. Of particular interest are the advances in a new type of switchable electrochromic windows: switchable mirrors. Switchable mirrors change their optical properties reversibly between reflective and transparent states, instead of between absorptive and transparent states. Proton-based and lithium-ion-based switchable mirrors have both been developed. Because switchable mirrors reflect sunlight and heat instead of absorbing, they are expected to remain cooler, more durable, and more energy efficient. In addition, they are less expensive with much faster deposition rates and less material requirement.
Monday Evening

May 11, 2009
Poster Session
4:30 p.m.-8:00 p.m. Poster-1 Tribological Properties of CrAlSiN Coating with Post-Deposition Heat Treatment

W.Y. Ho and C.W. Chen, MingDao University, ChangHua, Taiwan; and W.Y. Ho, National Pingtung University, Pingtung, Taiwan

Researches of (Cr, Al, Si)N coatings have confirmed that these coatings possess the excellent properties of the superhardness (~40 GPa) and thermal stability. The property changes of these hard coatings by a post-deposition annealing treatment are discussed in detail. In bulk materials, annealing treatments are well known and powerful practices to adjust their microstructure. However, the significance of heat treatments to optimize properties of (Cr, Al, Si)N hard coating for specific applications is new so far. In this study, a CrAlSiN coating was synthesized by cathodic arc deposition with Cr and Al88Si12 dual rotating cathodes. The post-deposition heat treatment was then applied to the as-deposited coatings. The microstructural changes of the coating before and after heat treatment was compared by X-ray photoelectron spectrooscope, X-ray diffractometer and scanning electron microscopy. The wear resistance of the coatings was studied by the ball-on-disc wear test without any lubricant. Field test of the forming dies with CrAlSiN coating with and without heat treatment was carried out. Results show that the post-deposition heat treatment in N2 gas may play a protective role to modify the structure and increase the tribological resistance of the CrAlSiN coating.

4:30 p.m.-8:00 p.m. Poster-3 Particle-in-Cell Monte Carlo Analysis of Inhomogeneities in Large Area Magnetron Discharges

M. Siemers, A. Pflug, and B. Szyszka, Fraunhofer Institute for Surface Engineering and Thin Films IST, Braunschweig, Germany

Due to the increasing demand on productivity and quality of magnetron sputtering coaters, the design of sputter sources with optimized homogeneity has been subject of extensive R&D efforts during the past decade. Substantial progress has been possible within the last few years where the increasing availability of parallel hard- and software architectures has made plasma simulation more applicable in the model based development of industrial plasma technology. With the use of massive parallelization we realized a full featured 3D simulation environment for low temperature plasma discharges based on the Particle-in-Cell Monte-Carlo (PIC-MC) approach. Complex geometries can be defined by a finite-element mesh structure as can be obtained from CAD data with a mesh generator. We present calculations on the erosion homogeneity of planar magnetrons focusing on the so-called "cross corner effect", i.e. regions with locally enhanced erosion located close to the target ends. The simulated erosion profile is in good agreement with experimental data obtained from target profilometry. The PIC-MC simulations reveal two mechanisms, high-energy electrons and plasma squeezing, which are mainly responsible for these deviations and which can be understood in the context of the three-dimensional magnetic confinement of the electrons. Furthermore, the total pressure dependency of these effects and their impact on the erosion profile on a substrate is investigated.
Monday, May 11
4:30 p.m.-8:00 p.m. Poster-4 The Latest Soft Electron Technologies

G. Gotzmann, F.H. Roegner, and O. Roeder, Fraunhofer Institute of Electron Beam and Plasma Technology FEP, Dresden, Germany

The Fraunhofer FEP in Dresden is part of the Fraunhofer-Gesellschaft, one of the largest R&D organizations in Europe. Since 1991 the FEP has been developing low-energy electron accelerators for innovative industrial applications: 1. Agriculture: Disinfection of seed products and also foods and animal feeds 2. Packaging: Sterilization of packaging for pharmaceutical products and foods 3. Medical technology: Sterilization of implants and instruments 4. Waste treatment: Deactivation of microbiologically contaminated waste. Seed products such as wheat and corn have traditionally been treated with toxic pesticides to kill any pathogens. An environmentally-friendly and effective alternative is now e-ventus® technology. This technology is being increasingly used in Europe and is being marketed by the EVONTA® Group. The pharmaceutical industry requires technologies for sterilizing a broad spectrum of products. In collaboration with Bosch®, innovative Advance-Beam technology has been developed for in-line sterilization of pharmaceutical packaging. Soft electrons are excellent for sterilizing and functionalizing medical components and instruments. The latest results in this area are presented.

Microbiologically contaminated waste (e.g. waste containing salmonellae, HIV (AIDS) and H5N1 (bird flu)) must be deactivated before disposal. In conjunction with Glatt®, the FEP has developed a process for treating liquid waste containing solid matter. The waste can also be granulated and refined into reusable materials.

Monday, May 11
4:30 p.m.-8:00 p.m. Poster-5 Cr-Si-N Coating for Aerospace Applications

E. Bousser, M. Benkahoul, M. Azzi, L. Martinu, and J.E. Klemberg-Sapieha, École Polytechnique de Montréal, Montréal, Canada

Erosion by solid particle impact is known to cause severe damage to critical components in aircraft engines. In order to find coating system solutions to this problem, we have undertaken a collaborative project with industrial partners to evaluate different functional coatings that could offer enhanced erosion resistance. While CrN coatings have not proved to be excellent erosion resistant coatings, they do offer excellent protection against corrosion. Therefore, in this work we have studied the Cr-Si-N coating system deposited on AISI SS410 substrates. We first present the material development phase, during which we deposited Cr-Si-N coatings with increasing silicon content on c-Si substrates. Then, following interface engineering, these materials were deposited on stainless steel at greater thicknesses. It was found that the addition of Si during deposition affects coating preferred orientation which in turn significantly influences mechanical properties at the microscopic level. Also, nitriding conditions were found to have a very strong impact on the corrosion resistance and adhesion. The pin-on-disk wear resistance was then evaluated and the best performance was obtained for the coating containing 2.3 at.% of Si with a wear rate 500 times smaller than that of the substrate. Finally, Solid particle erosion (SPE) testing was conducted according to the ASTM G76 standard with Al2O3 particles with an average diameter of 50 μm, a mean velocity of 70 m/s and an incident angle of 90°. The minimum erosion rate was found for Cr-Si-N with CrSi = 11.6 at.%. It is one order of magnitude lower than that of pure CrN and 20 times lower than that of the substrate. This high SPE resistance of the coating is attributed to an enhanced micro-hardness and an increased resistance to plastic deformation due to a dense (200) preferred orientation.
Monday, May 11

4:30 p.m.-8:00 p.m. Poster-6 Laser-Based Sensor for Real Time Sputter Monitoring and End Point Detection in Ion Beam Etch Systems

A. Yalin and L. Tao, Colorado State University, Fort Collins, CO; and N. Yamamoto, Kyushu University, Fukuoka, Japan

Real time in situ monitoring of the concentration of sputtered particles would provide a powerful tool for process control in ion beam etch systems, including end point monitoring. In this contribution we present a laser based sputter sensor that uses the continuous-wave cavity ring-down spectroscopy (cw-CRDS) technique. Cw-CRDS is a laser-based absorption diagnostic that provides ultra-high sensitivity by housing the measurement volume within a high-finesse optical cavity (placed within the vacuum chamber). The demonstrated system is based on detection of sputtered manganese atoms using a compact diode laser in the vicinity of 403.07 nm. The laser sensor is fully fiber coupled allowing integration to a variety of vacuum chambers and sputtering configurations. Measurements from a manganese-iron target are presented. End-point detection is demonstrated by monitoring the time dependence of manganese concentration for a multilayer target comprised of alternating manganese layers. Detection limits are shown to be adequate for today’s commercial ion beam sputter systems.

Monday, May 11

4:30 p.m.-8:00 p.m. Poster-7 Study on Surface Modification of Polycarbonate Polymer Plastics by Low Energy Ion Beam

Y. Yan, J. Wu, G. Zhang, Y. Wang, and P. Wen, Beijing Institute of Aeronautical Materials, Beijing, China

Ar/Oxygen ion beam generated in Kaufman ion beam source with low energy was employed for surface modification of Polycarbonate (PC) aimed at improvement of its surface statement. The water contact angle of modified surfaces was measured and surface composition as well as surface roughness were analyzed by X-ray photoelectron spectroscopy and atomic force microscopy. The results showed that the hydrophilicity of PC was significantly improved after modification because ion beam bombardment resulted in a large variety of reactive groups and then increased the oxygen content at the surface of PC rapidly. Surface roughness was also changed after modification due to ion beam etching effect. The rigidity, chemistry stability properties of the plastics were improved. And the optical property was changed slightly. The modification mechanism is discussed.
Monday, May 11

4:30 p.m.- 8:00 p.m. Poster-8 A Comparison of Deposition Techniques for High-End Optical Coatings

P. Biedermann, S. Wuethrich, and A. Jaunzens, Evatec Ltd., Flums, Switzerland

With its inherent flexibility, conventional vacuum evaporation is still the mainstay for commercial production of optical thin films. However, at relatively low coating flux energies of 1eV or less, there are limitations to the refractive index, optical and mechanical stability of the films produced. A number of enhanced evaporation techniques with higher flux energies, coating densities and improved optical indices have also found widespread where enhanced film properties are specified. Most recently, sputtering techniques have now shown themselves to be viable alternatives for mass production with the development of new sophisticated optical and plasma emission monitoring techniques. Reactive magnetron sputtering is now able to produce some of the highest performance optical films available with no spectral shift at increased temperature or humidity. However, the optical engineer needs to consider a number of factors in choosing a viable deposition technique for any particular application. New data from a number of evaporation and sputter techniques will be presented including a discussion on the relationship between film properties and microstructure.

Monday, May 11

4:30 p.m.- 8:00 p.m. Poster-9 Study of Structure Densification in TiO2 Coatings Prepared by Magnetron Sputtering Under Low Pressure of Oxygen Plasma Discharge

J. Domaradzki, D. Kaczmarek, and E.L. Prociow, Wroclaw University of Technology, Wroclaw, Poland; and Z.J. Radzimski, Silicon Quest International, Reno, NV

It is well known that the structure evolution of thin films prepared by reactive magnetron sputtering is strictly dependent on the particle energy at the film nucleation site. The energy could be changed typically by: (1) additional heating of the substrates, (2) decrease in plasma pressure, (3) increase in the temperature of the sputtered target surface (hot target) and (4) increase the sputtering power. Present work presents results of studies on structural and optical properties of the TiO2 thin films prepared by two methods: Low Pressure Hot Target Magnetron Sputtering (LPHTRS) and High Energy Reactive Magnetron Sputtering (HERMS). Both these methods allow adjusting the process energy using all four options mentioned above. In both processes oxide thin films are deposited from metallic targets using oxygen gas only instead of the usually used mixture of Ar-O2. Additionally, in HERMS, an increased amplitude of unipolar pulses powering the magnetron has been applied. It is shown that all prepared coatings were stoichiometric and by changing only the discharge voltage it is possible to influence the resulting structural phase and optical properties of prepared thin films. TiO2 thin films prepared using LPHTRS had anatase structure with refraction index n=2.1 (at 500 nm) whereas HERMS allows obtaining high temperature stable rutile structure with n=2.52 (at 500 nm), i.e. equal to the n value reported for the monolithic rutile. It is also shown that enhanced kinetic energy in HERMS caused higher degree of densification of the coatings and a change in the type of stress from compressive to tensile.
Monday, May 11
4:30 p.m.- 8:00 p.m. Poster-10 The Design and Development of an LPCVD Reactor for the Growth of 3C-SiC on Si

M. Orthner, L. Rieth, and F. Solzbacher, University of Utah, Salt Lake City, UT

A low-pressure chemical vapor deposition (LPCVD) reactor has been developed for the growth of beta-phase silicon carbide (3C-SiC) thin films on silicon. To our knowledge, this is the first LPCVD system that couples, by radiation, a resistive graphite heater with silicon substrates that are rotated from below. The heaters electrical, thermal, and mechanical properties were modeled and optimized using finite element analysis. The final design used a 6.5 inch (diameter) circular graphite serpentine heater with two individual current paths. Empirical measurements of substrate temperature agree well with FEA results and were made by melting a group of pure metals in vacuum. Thermal experiments when compared to numerical simulations are in good agreement, and demonstrate the system is capable of temperatures in excess of 1500°C with a uniformity of < ±10°C across the silicon substrates. Initial growth runs were performed with precursors: silane, propane, and hydrogen. X-Ray Diffraction (XRD) analysis found the microstructure of film was 3C-SiC with the observation of a reflection at 41.4° (0/20).

Monday, May 11
4:30 p.m.- 8:00 p.m. Poster-13 Optical Emission Spectroscopy of Ni, Cr, and NiCr 80/20 for DC and High Power Impulse Magnetron Sputtering (HiPIMS)

H. Gerdes, J. Wellhausen, R. Bandorf, and G. Braeuer, Fraunhofer Institute for Surface Engineering and Thin Films IST, Braunschweig, Germany

In technical applications, applied forces are often measured by polymer strain gauges. Unfortunately, those strain gauges are also highly sensitive to humidity and temperature resulting in creep and swelling. By direct application of the strain gauges onto the surface of the workpiece by physical vapour deposition process (PVD) this negative influence is avoided. As typical material for a stress sensing thin film layer, NiCr is well established. Since the sensing properties of these layers are influenced by the plasma properties, e.g. the induced power or the PVD process itself (DC or HiPIMS), using High Power Impulse Magnetron Sputtering HiPIMS, the induced peak power and current is significantly increased and therefore the material properties are also modified. For a better understanding of the influence of the process used on the plasma properties, optical emission spectroscopy was used for investigation. In this paper the influence of pulse length and charge voltage of the pulse unit on the resulting plasma was investigated. As reference also OES spectra from DC processes obtaining a comparable average power are used.
Monday, May 11

4:30 p.m.- 8:00 p.m. Poster-14 Decorative Coating Deposition by PVD

J. Esparza, R. Rodriguez, J.A. Garcia, and M. Rico, Asociacion de la Industria Navarra, Pamplona, Spain

The development of the Physical Vapor Deposition (PVD) technique has traditionally been linked to machine tool applications. In the last decades, researchers have been working in several new applications for PVD coatings such as photovoltaic technology, medical applications or decorative coatings. The versatility of the PVD technique provides a wide range of colors and optical effects that could be very interesting in order to increase the added value of some specific products. The objective of this study is to deposit different PVD decorative coatings on several substrates such as tiles, metals, glass or even polymers. In addition, the characterization of the mechanical properties of this new coatings has been performed. The results of three research lines are presented: The first study covers the simulation of a specific color (nickel) on brass substrate, the control of the shade of the coating depends on several parameters such as temperature, pressure, targets, bias and intensity. In the second research line, several colors have been obtained by the deposition of titanium oxides, the color depends on the thickness of the oxide layer. In the last research line, the result of the deposition of metallic and ceramic PVD coatings on different substrates is presented.

Monday, May 11

4:30 p.m.- 8:00 p.m. Poster-15 Research of Electrical, Optical and Structural Characteristics of Ga-Doped ZnO Coatings Deposited by Magnetron Sputtering on a Polymeric Substrate

M. Misels-Piesins, E. Machevski, I. Ashmanis, and V. Kozlov, Sidrabe, Inc., Riga, Latvia (Presented by E. Yadin, Sidrabe, Inc., Riga, Latvia)

While Al-doped ZnO films on glass have been recently brought into production as a low-cost TCO for silicon solar cells, they are not applicable to plastic substrates due to elevated deposition temperatures. A promising low-cost TCO for flexible substrates is Ga-doped ZnO (GZO). The electrical properties of this material are highly dependent on the process parameters, therefore, it needs thorough investigation and optimization of the deposition technology. In this work, smooth GZO films were deposited from ceramic targets on PET substrates by DC and MF sputtering in a roll-to-roll coater. Influence of working pressure, oxygen, hydrogen, sputtering power, substrate temperature, magnet system configuration, silicon dioxide buffer layer, substrate angle and electrical shielding of charged particles were investigated. Depositions at different parameters were performed without interrupting magnetron operation to ensure repeatability. PET substrate was substituted with glass to evaluate characteristics before and after thermal annealing in oxygen-free ambient. Statically deposited samples indicated a strong tendency to form poorly conducting regions directly above the erosion zones. Within the optimal process window, a typical resistivity of 2mOhm-cm and optical transmittance of up to 85% in the visible spectra was repeatedly achieved.
Monday, May 11

4:30 p.m.- 8:00 p.m. Poster-16 The Tribological Characteristic of TiN, TiC, TiN/TiC Films Prepared by Reactive Pulse Arc Evaporation Technique


Titanium nitride (TiN), titanium carbide (TiC) thin films and TiN/TiC multilayers have been deposited on AISI 304 steel substrates by PAPVD - Reactive Pulsed Arc method and characterized from the structural, mechanical and tribological point of view. The structural characterization of the coatings was performed by XRD showing a preferential orientation in the (111) diffraction peak, which is characteristic of the FCC phase in this kind of coatings. Tribological behavior was investigated using Ball on Disc technique (Nanovea) with an Al₂O₃ ball, 10 cm/s speed, distance 100 m and 1N load. The average COF was measured, showing mean values of 0.64 for TiN and 0.53 TiN/TiC. A dynamic wear curve was performed for each coating, observing the evolution and wear mechanism as a function of the distance, using optical stereoscopic techniques.

Monday, May 11

4:30 p.m.- 8:00 p.m. Poster-17 The Effect of Deposition Temperature on Tribological Behaviour of Ti-Al-N Coatings Deposited by Magnetron Co-Sputtering Technique

M. Cano, J. Restrepo, A. Ruden, and F. Sequeda Osorio, Universidad del Valle, Cali, Columbia; and J.M. Meza, Universidad Pontifica Bolivariana, Medellin, Colombia

The Ti-Al-N coatings have been deposited on H-13 tool steel substrates and silicon wafers by DC Magnetron co-sputtering at different temperatures (50°C, 150°C and 200°C), in order to study the influence of this parameter on the tribological behavior. For all coatings a crystalline NaCl structure was obtained which is typical for titanium based nitrides. For all coatings a strong (200) texture relative to the (111) orientation was found. Profilometry measurements showed a decrease of average surface roughness (50 to 28 Angstroms) as deposition temperature was varied from 50°C to 200°C and nanoindentation measurements (Nanovea Instrument) indicated a maximum hardness of 23Gpa. Tribological measurements were made using a room temperature tribometer (pin/ball on disk, CSEM Instrument) allowing measurement of the dependency COF on cycles (sliding distance). The evolution of COF with the cycles was measured under different conditions: sliding speed (10 and 20 cm/s), load (1N and 3N) and wear rate of the ball (WC, 100Cr₆ and Al₂O₃) and coating. The wear tracks were examined by optical methods and SEM, in order to identify wear evolution and corresponding mechanisms (mild wear, fracture or delamination). The results showed that the load and sliding speed produces changes in the COF and wear mechanism. In coatings deposited at 150°C and using WC balls and low speed (10cm/s), the COF was 0.85 and the debris is present in all wear track. When increased speed (20cm/s), the COF decreases (0.49) and debris was produced just at the last 1000 cycles and the wear rate decreased 50%. Similar behavior was observed when using other counterparts, balls materials.
Monday, May 11
4:30 p.m.- 8:00 p.m. Poster-18 Tribological Properties of Duplex TiN Coatings Applied on Chrome Based Steels

A. Murcia, Universidad del Valle, Cali, Columbia; S.P. Bruhl, Universidad Tecnologica Nacional, Concepcion de Uruguay, Argentina; A. Neira, North Carolina State University, Raleigh, NC; and F. Sequeda Osorio and A. Ruden, Universidad del Valle, Cali, Columbia

Duplex process consisting on plasma nitriding \([\text{N}_2 + 3\text{H}]\) and Titanium Nitride- TiN PVD coating deposited by Magnetron Sputtering, was conducted on AISI 420 substrates, showing to be effective in reducing friction coefficient (COF) and wear rate and increasing surface hardness. Chrome based steels are widely used due to its corrosion resistance, high tensile strength, moderate corrosion resistance and the high load-carrying capability. Tribological properties were determined by ball-on-disc test, while the wear mechanism against an alumina ball was assessed by analyzing the wear debris using SEM. Results showed a 20-25% decrease in COF and wear rate as compared with steel substrates with no duplex treatment. Micro hardness analysis showed an increase in surface hardness of 50%. Low hardness decrease due to indentation depth was also observed through nano-indentation measurements. Using SEM and XRD, the effect of nitrogen inclusion on the crystal structure of the steel was further analyzed as well as the interface between the nitride layer and the hard coating.

Monday, May 11
4:30 p.m.- 8:00 p.m. Poster-19 Study of Synergistic Effect of Erosion-Corrosion of CrN and TiN Hard Coatings on AISI 1045 Mild Steel

H. Payan, W. Aperador, F. Sequeda Osorio, and A. Ruden, Universidad del Valle, Cali, Columbia

Results of the study of synergistic effect of corrosion-erosion of CrN and TiN hard coatings deposited by PVD - Magnetron Sputtering on AISI 1045 mild steel and a comparison with a stainless steel AISI 316 and a mild steel AISI 1045 with no coating are shown. These behaviors have been investigated by potentiodynamics measurements (Tafel curve) in a solution of 0.5M \(\text{H}_2\text{SO}_4 + 3.5\%\) in weight of \(\text{NaCl}\) and mass loss techniques respectively. Results of electrochemical testing indicate the behavior of CrN and TiN are quite different. For different angles of incidence of corrosion-erosion fluid, it was observed that part of the layer of CrN was stripped from the substrate due to the action distributed by the continuous shocks of the abrasive particles; however the electrochemical behavior shows that CrN was the noblest material in respect to the solution and presents a smaller current density than TiN, indicating better corrosion resistance. This is possibly due to CrO formed on the coating surface of CrN being more protective and corrosion resistant than those of the TiN. This can also explain why CrN has a similar behavior to the stainless steel. Synergistic effect in all the studied materials are presented and discussed.
Monday, May 11

4:30 p.m.-8:00 p.m. Poster-20 Plasma Analysis of a Novel PECVD Process for Corrosion Resistant Interior Coating of Pipelines

S. Lapp and F. Placido, University of the West of Scotland, Paisley, United Kingdom

The main problems of industrial piping are corrosion and erosion/wear of the interior surface. A low-priced and effective coating technology to produce a hard and smooth film without any pinholes would reduce cost of plant construction and maintenance in a wide area of industry. We report on work on characterizing a novel DLC coating technology to reduce corrosion inside of pipelines and improve flow rates of fluids. The pipe itself acts as the vacuum chamber where the molecules of the process gases are decomposed by the ignited pulsed dc plasma inside the pipe. This novel concept allows the interior coating of parts with different dimensions and geometries. Optical emission spectroscopy (OES) is used to analyze the emitted light of the plasma to characterize the plasma condition inside the pipe at different positions along the pipe. The neutral density and ion density of the different process gases can be determined to understand the respective transition for different process conditions. The electrical properties of the plasma such as electron density, ion density and electron energy distribution function (EEDF) were analyzed by a Langmuir Probe plasma diagnostic system. The set up used allowed a spatial and time resolved investigation of the plasma condition along and across the pipe. The presentation contains the analysis of the plasma conditions in pipes of variable size and for different process parameters like power, pressure and gas flow.

Monday, May 11

4:30 p.m.- 8:00 p.m. Poster-21 The Effect of Superfinishing and Plasma-Assisted PVD and CVD Coatings on Rolling Element Bearings Under Lubricant Starvation Conditions

J. Eichler, University of Sheffield, Sheffield, United Kingdom; G. Doll, Timken Research, Canton, OH; A. Leyland and A. Matthews, University of Sheffield, Sheffield, United Kingdom

This is a report on tests carried out using a bespoke high-cycle rolling-contact test facility based on a thrust ball bearing configuration. The test machine is instrumented for temperature, torque, vibration and speed measurement. Following on from previous publications, this report concentrates on rolling contact fatigue behaviour of coated and uncoated counterfaces under boundary lubrication conditions created by inadequate lubrication. The main coatings tested were Cr₂N and WC/aC:H which were deposited by plasma-assisted PVD and hybrid PVD-CVD processes respectively. In addition, the effect of vibratory superfinishing, both in isolation and as a surface pre-treatment, is investigated. Data is presented illustrating the benefits provided by surface modifications under these extreme operating conditions and their ability to delay the onset of catastrophic bearing failure.
Heuréka!
Monday, May 11

7:30 p.m. H-1 Patents on Plasma and Surface Engineering: Actual Trends in Patents, a Patent Database Study

R. Bethke, Fraunhofer Institute for Surface Engineering and Thin Films IST, Braunschweig, Germany; B. Rager, Fraunhofer-Gesellschaft, Muenchen, Germany; and W. Diehl, Fraunhofer Institute for Surface Engineering and Thin Films IST, Braunschweig, Germany

R&D is the key driver of innovation and an essential part to protect these developments is patent protection. Patent shield is both, a security against other competitors and an opportunity to seize market exclusivity for a novel product or treatment. The amount of patents on Plasma and Surface Engineering is certainly large and properly unstructured. Currently there is no data base regarding patents on Plasma and Surface Engineering available. To get global and detailed patent information a data base was developed for patents (World Wide) regarding Plasma and Surface Engineering. It was the aim to analyze the patents concerning global results like timeline, major designated countries, or applicants. Additionally, detailed patent analyses are possible. In the first part of the talk the methodology, the utilized databases as well as the analyzing tools will be explained. In the second part some results will be presented. Global patent activities relating to Plasma and Surface Engineering based on patents and applications, published from 1988 to 2008, were examined. The study presents key and emerging companies, universities and R&D-organizations, as well as historical and technological trends of patent activities. Furthermore, this lecture shows the results of geographic analysis and the separation in technology fields regarding different plasma applications.

Monday, May 11

8:00 p.m. H-2 Voltage Control for Reactive Sputtering: Achieve Up to 10 Times the Typical Sputter Rate While Dramatically Reducing Input Power Requirements


In today’s large-area thin-film vacuum-coating applications, vacuum pumps and power supplies typically consume the most energy. This is especially true for reactive processes. The best way to reduce incoming power requirements is to run high on the transition curve, but traditionally, this is very expensive and complicated. This presentation describes a simple solution that significantly reduces input power consumption, while dramatically increasing sputter rate: a power-supply voltage control feature. Although with its use, certain process issues must be addressed, voltage control is a simple method for achieving significant cost savings and high-quality, repeatable films.
The low ion energy and high current flux of broad-area gridless end-Hall ion sources can have significant advantages over high-energy gridded or gridless ion source systems when processing glass and engineering thermal plastics. Specifically the lower energy spectrum of such ion sources are shown to improve surface energy and adhesion with limited process exposure times and while inducing very limited optical, thermal or sub-surface layer damage which leads to mechanical embrittling, deformation and optical yellowing. We examine fundamental surface treatment properties of multiple materials (glass, silicon, polycarbonate, thermoplastic polyurethane, polytetrafluoroethylene, and polyethylene terephthalate) when processed under identical conditions in pure oxygen and argon-oxygen blends using contemporary end-Hall ion source technology. Surface energy, optical transparency, substrate thermal damage and FTIR spectral data of treated surfaces are reported along with adhesion performance of PVD sputtered titanium films. Optimal reduction in surface wettability is noted in all materials with the inclusion of 10-30% of oxygen with little to no change in optical transparency. In addition to these results, typical treatment rates are deduced against ion beam current dosage to help process developers scale end-Hall ion source systems to various wide-area batch and in-line vacuum treatment applications.

Transparent films are playing an increasingly important role in modern industry because films are critical components of many high technology industries such as photovoltaics, medical implants, thin-film batteries, flexible displays, advanced semiconductors and MEMS. Film designers are providing tightly tolerated designs to manufacturers and they, in turn, are challenged to consistently meet the required quality in various applications. In order to get the best product, however, proper film inspection is a key requirement. Ellipsometers can measure film thickness at a single spot or over a small field of view. Stylus techniques can inspect the thickness on a line-by-line basis as long as an edge is available. Three dimensional inspection techniques are increasingly required to control the process over large areas and provide both surface and film thickness information from a single system. In this presentation, interference techniques for film metrology using white light will be described. The technique provides 3D top surface, interface and thickness profiling. For thick (above 2μm) and thin film, different signal analysis techniques are required. Various measurements will be presented illustrating the capabilities of the technique and the needs of several applications spaces discussed.
A new method of generating pulsed cathodic arc discharge was developed. In order to ignite and sustain the arc discharge, a voltage pulse output with sharp voltage oscillations was applied between cathode and anode. The typical pulse duration was in the range of 500-3000 µs. During the experiment, it was found that the growth of the arc current can be controlled by changing the voltage rise time and the amplitude of these oscillations. By applying different voltage oscillations in a single pulse, it was found that the arc discharge current can be controlled. OES spectrums for pulsed arc discharge with aluminum, titanium/aluminum, and carbon targets in either pure oxygen or oxygen/argon atmospheres will be presented. Voltage and current waveforms with different voltage oscillations will be discussed. SEM images of aluminum oxide films deposited with voltage a pulse duration of 1000 µs will be presented. By controlling the voltage rise time of the voltage oscillations in the beginning of the pulse, a new mode of arc discharge for titanium/aluminum target (50/50)% was observed. This new method increases ionization of the evaporated material and should help to control the size of macro particles when compared with conventional continuous arc discharge.
Joint Session on Atmospheric Plasma Technologies
Rare gas discharges sustained at atmospheric pressure are affected by the phenomena of contraction (reduction of the discharge volume) and filamentation (breaking of a contracted plasma channel into two or more filaments). Contraction has been observed in DC, RF and microwave discharges while filamentation is specific to microwave discharges. Reducing or eliminating contraction and filamentation is essential in the optimization and development of some industrial applications using high pressure discharges. Recently, it was shown that it is possible to achieve non-contracted and non-filamentary microwave discharges at atmospheric pressure. A contracted/filamentary discharge in a given pure rare gas at atmospheric pressure expands/homogenizes radially upon addition to it of a specific small amount (generally less than 1%) of another rare gas having a lower ionization potential. The characteristics of contracted and expanded discharges at atmospheric pressure are described and the mechanisms causing discharge contraction as well as expansion analyzed. Radial contraction is due to the influence of non-uniform gas heating on molecular ion kinetics while the discharge expands because the charged particle-loss is governed by atomic-ion diffusion. This last fact by itself supports the essential role of molecular ions in discharge contraction.

Plasmas have been used widely for the pre-treatment of metals prior to adhesive bonding applications. In this study the use of a plasma polymerised primer layer as a means of enhancing the adhesion of siloxane polymers to steel is investigated. The primer layer consisted of a mixture of tetraethoxysilane (TEOS) and polyhydrogenmethyl siloxane. This liquid mixture was nebulized into a helium atmospheric pressure plasma jet. The resulting primer coating was characterised using x-ray photoelectron spectroscopy (XPS), Fourier Transform infrared spectroscopy, optical profilometry, water contact angle, atomic force microscope and spectroscopic ellipsometry. The adhesion of the siloxane elastomer was accessed using 45° peel strength measurements. Among the parameters investigated was the influence of the atmospheric plasma jet to substrate distance on primer performance. In addition the effect of primer thickness was studied by systematically altering the number of passes of the jet over the steel substrate. The thickness of the primer investigated ranged from 50 to 600 nm. The influence of time after primer deposition on subsequent metal to siloxane elastomer adhesion was investigated up to a period of 14 days. Over a 15-fold enhancement in the adhesion of siloxane elastomer to stainless steel was achieved using the plasma polymerised primer layer.
9:10 a.m. JAPT-9 Equipment for Large Area Plasma Processing at Atmospheric Pressure

I. Dani, G. Maeder, J. Roch, P. Grabau, B. Dresler, D. Linaschke, S. Tschoecke, S. Kaskel, and V. Hopfe, Fraunhofer Institute for Material and Beam Technology IWS, Dresden, Germany

Atmospheric pressure plasma technologies offer a unique combination of technological and economic advantages. For large area processing, two types of plasma sources are presented: a linearly extended DC arc discharge and a microwave plasma source. The DC arc plasma source offers a scalable working width of up to 350 mm at the moment. The second source is a scalable microwave plasma source for atmospheric pressure operation. Plasma gases are mainly mixtures of nitrogen and argon with reactive gases. PECVD reactors for inline air-to-air processing of flat substrates up to 156 mm wide for deposition of SiO₂, a-C:H, and SiNₓ:H as well as a flying coater head for scanning the surface of larger substrates are presented. Also, equipment for continuous atmospheric pressure plasma-chemical etching of silicon was developed. All reactors include purge gas curtains to assure an inert working atmosphere, enabling the deposition of non-oxide films, and to prevent the leakage of reactive gases. Fluid dynamic calculations have been used to optimise the reactor and plasma source design. For process characterisation optical emission and in situ FTIR spectroscopy are applied. Dynamic deposition rates are up to 25 nm/min, rates for silicon etching can be 10 times higher.

9:30 a.m. JAPT-10 Current and Future Prospects of Non-Thermal Plasmas Exhaust-Air Pollution Control

R. Brandenburg, R. Basner, and K.-D. Weltmann, INP Greifswald e.V., Greifswald, Germany

In our highly industrialised society, the reduction of emissions from exhaust gas streams is becoming more and more important. Due to its impact on air, soil and water, exhaust pollution affects the environment and thus human health. Therefore environmental norms and standards are constantly increased by national and international authorities. The possibilities of air-pollution control by means of non-thermal plasmas are well known. As ionised gases, plasma contains active and highly reactive species, in particular radicals or oxidizing compounds, which can decompose pollutant molecules or organic particulate matter (e.g. soot). However, the applicability of non-thermal plasma processes has been turned out to be limited, too. It is determined by many facts, e.g. energy costs or formation of by-products. Under this aspect, hybrid processes combining non-thermal plasma with other technologies such as catalysis, absorbed agents or wet processing become a focus in recent research activities. The contribution shall summarize the possibilities of non-thermal plasma exhaust-air pollution and discuss its prospects and limits. Furthermore, it will report on commercially available plasma based and plasma assisted processes as well as discuss current trends and concepts in research.
Tuesday, May 12

9:50 a.m. JAPT-11 Environmental Applications of the Atmospheric Pressure Plasma Sources

H. Baránková and L. Bárdos, Uppsala University, Uppsala, Sweden

Non-equilibrium atmospheric plasma sources based on the hollow cathode, microwave and hybrid concepts were developed for conversion of hazardous gases and reduction of particulate matter in the exhaust gas. As these applications require a stable performance especially in molecular gases, the design of plasma sources must be such as to level off instabilities associated with molecular gases. The results on the cleaning process are presented, together with experimental results on discharge generation which formed a basis for the plasma reactor designs. The gas conversion plasma reactors have diverse applications and can be for example integrated into PE CVD atmospheric pressure systems.

Tuesday, May 12

10:30 a.m. JAPT-12 Atmospheric Pressure Plasma Deposition of Transparent Conductors - Tailoring Precursor Chemistries

K. Johnson, S. Jha, R. Sailer, and D. Schulz, North Dakota State University, Fargo, ND

Atmospheric-pressure plasma (APP) processes such as corona discharge have demonstrated utility in the cleaning steps associated with roll-to-roll manufacture. APP has more recently found applicability in the modification of surface energies given the ability to form plasma-polymerized coatings from precursors that contain an olefin functional group. The application of APP deposition to materials relevant to flexible electronics requires the use of functional precursors that maintain the following characteristics at 760 Torr: (1) a significant vapor pressure; (2) ability to be transported to the reaction zone without decomposition; and, (3) ability to form the targeted phase with byproducts that are eliminated from the growth surface. We have been investigating the utility of metal-organic complexes as precursors to transparent conducting oxides toward web-based manufacture of photovoltaics, flat panel displays, etc. In this paper we will describe our efforts toward APP deposition of In-Sn-O using Sn(II) and In(III) beta-diketonate complexes. Helium carrier gas, O₂ reactant gas and growth temperatures from ambient to 300 °C have been evaluated with 20 minute growth cycles providing 50-100 nm thick coatings over 30 cm². The as-deposited films exhibit light transmittance in excess of 90% over the visible spectrum while maintaining resistivities on the order of 10⁴ ohms/cm.
Optical Coating
8:30 a.m. O-1 Monte Carlo Analysis of Random Thickness Errors in Infrared Optical Coatings

D. Fuller and W. Hasan, FLIR Systems, Inc., Wilsonville, OR

Infrared imaging systems commonly employ an assortment of lenses, mirrors, beam splitters, and filters. Each of these elements utilizes optical thin films to make them functional. As is the case with most optical coatings, layer thickness of infrared coatings is critical to spectral performance. Manufacturing errors result in non-optimum spectral performance and loss of product if errors are excessive. This paper utilizes Monte Carlo error analysis to predict production yields of infrared optical coatings. Statistical spectral data is presented based on repetitive calculations (e.g., 100,000 iterations). Two case studies are presented: 1) a long-wave infrared band pass filter, and 2) a mid-wave infrared antireflective coating. Empirical results are compared to statistical predictions. The effects of coating design on error sensitivity are discussed. Comparative coating designs are presented which offer similar spectral performance, but different sensitivity to thickness errors.

8:50 a.m. O-2 Modern State of Art in Design and Monitoring of Optical Coatings

A. Tikhonravov and M. Trubetskov, Research Computing Center, Moscow State University, Moscow, Russia

Invited 40 min. Talk

In recent years a tremendous progress in design of coatings of various types has been achieved. Non-local design techniques based on the needle optimization, Fourier-transform, inhomogeneous refinement enable an optical coating engineer to design two-component multilayers, rugate and quasi-rugate coatings with quite complicated spectral characteristics. In many situations multiple theoretical designs with excellent spectral properties can be obtained. These multiple designs can have different structural properties and thus more opportunities for choosing the most practical design are provided. For example it is possible to design hot mirrors having only thick layers at an expense of some increase of design total optical thickness. As it was shown at two last Optical Interference Coatings meetings in Tucson, new opportunities are provided by designing of multilayers containing very thin transparent metal layers. Modern optical coatings may have many dozens of layers and their successful practical implementation is very dependent on accuracy of monitoring of thicknesses of deposited layers. In recent years direct optical monitoring of coating production has gained more attention all over the world. This relates both to monochromatic and broadband monitoring techniques. In the case of direct optical monitoring layer thicknesses are monitored on one of the manufactured samples and this is an obvious advantage of direct techniques. However, a strong cumulative effect of errors in layer thickness is often observed when coatings with several dozens of layers are deposited using these techniques. In the last years several different monochromatic monitoring approaches aimed at reducing cumulative effect of thickness errors have been proposed. Recently presented theoretical results show that broadband optical monitoring is much less critical to measurement errors than monochromatic optical monitoring. This makes broadband monitoring especially attractive but more efforts are now required for developing broadband techniques with reduced cumulative effect of thickness errors.
Tuesday, May 12

9:30 a.m. O-3 Designing Optical Coatings by Using Low-Index Equivalent Layers and Low-Index Effective Media

U. Schulz and N. Kaiser, Fraunhofer Institute for Applied Optics and Precision Engineering IOF, Jena, Germany

The lowest refractive index material exhibited by a naturally available material is about 1.38 (magnesium fluoride). For some applications, especially broadband antireflective coatings for low-index substrates (n=1.5) it would be useful to have thin films with a lower index. The effect of a single layer with an equivalent refractive index below the index of natural materials can be achieved in a limited wavelength range by applying special designed layer sequences. On the other side, porous or structured layers can act as effective media with very low refractive index. This paper presents some designs showing the effect of both types of low-index replacements on examples.

Tuesday, May 12

10:30 a.m. O-5 Using Different Thin-Film Design Software for Different Requirements

U. Schallenberg, mso jena Mikroschichtoptik GmbH, Jena, Germany

Invited 40 min. Talk

This presentation gives a brief overview on the use of commercial thin-film software for the design and the assistance of manufacturing thin-film optical coatings, particularly with regard to practical questions and special problems. With the means of well-known thin-film coating types, it is exemplified which performances optical coatings may bring into an optical system, which theoretical formalism is required to describe these performances, and which components for manufacturing and characterizing of these coatings are required. Packages are listed and briefly described that provide easy manageable program tools for design, manufacture assistance and characterization. It is shown which software packages have to be used necessarily to fulfill the different requirements given by high performance thin-film coatings for broadband antireflection, non-polarizing beam splitters, wide blocking band-pass filters, and steep-edge filters. In addition, software tools for the optical monitoring during the deposition and for some re-engineering problems after the deposition are demonstrated. Finally, some tools are discussed that can be recommended for an efficient serial production according to the state-of-the-art of thin-film deposition technologies.
11:10 a.m. O-4 Broad Band Metal Dielectric Filters

D. Cushing, University of Arizona, Tucson, AZ

Filter designs with bandwidths greater than 30 nm for wavelengths in the visible spectrum will be described. Both reflective and transmissive types will be compared for use in optical systems with large fields of view. The filters will contain 7 to 45 layers consisting of two refractory oxides and a multiplicity of metal layers to provide the desired spectral results. The filters are designed for image quality applications. The pros and cons of reflective versus transmissive types will be discussed.

11:30 a.m. O-7 Computer Simulation of Monitoring of Narrow Bandpass Filters at Non-Turning Points

R. Willey, Willey Optical Consultants, Charlevoix, MI; and A. Zoeller, Leybold Optics GmbH, Alzenau, Germany

The present work is the result of combining the work reported by Zoeller, et al. on the computer simulations of coating processes with monochromatic monitoring together with the work reported by Willey on the design and monitoring of narrow bandpass filters at non-turning points. A new software tool which simulates the coating process with monochromatic optical monitoring was introduced which can deal with systematic and random errors of thickness, deposition rate, refractive indices, etc. Narrow bandpass filters have historically been designs of quarter waves at the passband wavelength, and have been monitored at the turning points using the passband wavelength. By direct monitoring at the passband wavelength, errors have been shown to be primarily self compensated, and have allowed much better performance than could otherwise be expected. The turning points are difficult to detect precisely and accurately because the change in transmittance with thickness becomes zero at the desired termination point. The new simulation program is applied here to designs which have been adjusted to have all layer terminations at a distance from turning points in order to gain greater change in the reflectance or transmittance with thickness and thereby reduce layer termination errors. The simulation has allowed the assessment of the merits of the non-turning point monitoring and its limitations as compared to the conventional approach.
Stress is a key factor causing deformation of coated optics. It is important to develop in-process observation technology in optical coating deposition system, and to control stress status more effectively and efficiently. In this paper, an instrument for \textit{in situ} stress measurement in an optical coater based on optical deflection of two parallel light beams is introduced. The stress evolution of thin film can be observed in process. The instrument is applied to investigate the deposition of single-layer and multilayer coatings in different conditions. Results show that HfO$_2$ films have a stable tensile stress of 360MPa–660MPa for all deposition conditions. The tensile stress becomes larger when the vacuum is higher. SiO$_2$ films show a stable compressive stress of -350MPa.
Large Area Coating
Tuesday, May 12

8:30 a.m. L-1 Energy Saving Glass Solutions

R. Blacker, Guardian Industries Corporation, Carleton, MI

*Invited 40 min. Talk*

In 2007, the global demand for advanced flat glass products was $22.3B, split primarily between Europe (29%) and North America (25%), with China, Japan and rest of world making up the remainder. In the US alone, this volume is equivalent to 5.9Bft² of flat glass sales. Architectural glass currently accounts for 16% of this volume, with demands forecast to rise 3.4% per annum to $5.8B in 2012. Solar control glass will account for only 36% of this demand; the remaining 64% being available for insulation upgrade to reduce energy usage in buildings. Currently in the US, windows consume up to 30% of the heating and cooling requirements for buildings; equivalent to 4.1 quadrillion BTU's (quads). The National Fenestration Research Council (NFRC) has estimated that between 1983 and 2005, the implementation of energy saving low-e windows has yielded direct energy savings of 6.1 quads. The NFRC estimates that such savings can be further maximized, and that the current 4.1 quad window energy consumption can be reduced to below 1 quad. This may be achieved by implementing new glazing technologies that employ window units with very low U-values, dynamic glazing able to modulate transmission and solar gains, and heated glazing that maximizes occupant comfort. This paper identifies and describes these and other technologies that are able to maximize energy savings and increase consumer comfort levels in both commercial and residential applications.

Tuesday, May 12

9:10 a.m. L-2 Enhanced Growth of Thin Silver Films via HiPIMS Deposition

G.T. West and P.J. Kelly, Manchester Metropolitan University, Manchester, United Kingdom

Thin silver films are commonly deposited via conventional DC magnetron sputtering for use in a variety of applications, including low-emissivity coatings, which require high transparency to visible light and good electrical conductivity. To achieve these properties the films need to be fully dense, although the typical growth mechanism is governed by the formation and coalescence of island structures leading to the generation of voids and uneven film surface topography. The result is that for acceptable electrical properties, transparency is often compromised due to the excessive film thickness required for continuous conductive layers. The HiPIMS (high power impulse magnetron sputtering) process delivers a highly ionised deposition flux with the potential to enhance adatom mobility without the need to apply any substrate heating or electrical bias, which can be problematic for large-area dielectric substrates such as float glass or polymer web. Thin silver coatings were deposited via continuous DC sputtering and HiPIMS at identical time-averaged power, in order to compare the structure and growth mechanisms via techniques including SEM and XRD. HiPIMS operating parameters (in particular the peak pulse power) were varied to optimise film growth and hence the resulting optical and electrical properties of the coatings.
Tuesday, May 12

9:30 a.m. L-3 Sensors for Uniform Reactive Magnetron Sputtering Deposition on Large Areas

V. Bellido-Gonzales, B. Daniel, D. Monaghan, Gencoa Ltd., Liverpool, United Kingdom; and J. Counsell, J. Counsell Ltd., West Kirby, United Kingdom

Reactive magnetron sputtering is a very common industrial coating deposition technique. Reactive magnetron sputtering occurs whenever the deposited chemical composition of the coating substantially differs from the composition of the magnetron sputtering targets (e.g. TiOx coatings deposited from Ti targets). Up to a certain point, magnetron sputtering has been a tolerated necessity. Industry has “opted-out” on certain reactive sputtering processes in order to gain process stability and reproducibility. The use of compound targets has already been adopted, e.g. ITO, ZnO:Al, and more recently TiOx. Most of these compound targets still would require a certain degree of reactive sputtering, especially for large area deposition requiring high coating uniformity. One of the key elements in maintaining a uniform reactive sputtering process over a large area under control is the sensor or sensors which are used as reference in the closed loop control. The lack of stable sensors and/or misuse or misplacement of sensors, has led the industry in many instances to take safer options. The present paper would try to highlight the current sensor availability with its pros and cons in large area uniform reactive magnetron sputtering deposition.

Tuesday, May 12

9:50 a.m. L-4 Properties of Plasma Polymerized Thin Films Deposited from Hexamethyldisiloxan (HMDSO) by Magnetron-PECVD Process

R. Nyderle, R. Bluethner, and T. Preussner, Fraunhofer Institute for Electron Beam and Plasma Technology FEP, Dresden, Germany; and D. Pavic, Bluetec GmbH & Co. KG, Trendelburg, Germany

HMDSO plasma polymer films are known to reduce the surface free energy and the permeation rate of water vapor. In this work results are presented from the investigation of surface properties of plasma polymerized films deposited from hexamethyldisiloxan (HMDSO) by magnetron-PECVD process. A rotatable dual cathode arrangement has been used as plasma source. Optical properties, deposition rate and film composition of these plasma polymerized films have been evaluated with respect to their application as top coating on solar thermal absorbers.
Tuesday, May 12

10:30 a.m. L-5 Thickness Dependence of Electron Transport in Polycrystalline SnO$_2$:F Films

T. Ikeda, H. Odaka, and T. Oyama, Asahi Glass Co., Ltd., Kanagawa, Japan

The electric transport in polycrystalline SnO$_2$:F thin films grown by atmospheric pressure thermal chemical vapor deposition (AP thermal CVD) has been studied. The samples with thick SnO$_2$:F film were ground by diamond paste to reduce the thickness to different levels for investigation of thickness dependence of the electrical property. From Hall measurement on the samples with different thickness, it was clear that the electron mobility increased with film thickness together with grain size. Petritz model for layered material was introduced to describe the mobility in each layer and the modified Seto model for a degenerated semiconductor was used to determine the grain boundary barrier height for electron transportation. Optical mobility calculated from reflectance spectra in the IR region using Drude model was compared with the Hall mobility. The electron transport in SnO$_2$:F will be discussed based on these results.

Tuesday, May 12

10:50 a.m. L-6 Spatio-Temporal Measurements of Plasma Properties in an AC Magnetron Source Using an Automated Boxcar Langmuir Probe

P. Greene and P. Brillhart, Applied Materials, Fairfield, CA; and B. VornDick and S. Shannon, North Carolina State University, Raleigh, NC

Several plasma parameters have been measured as a function of time and location in a rotating dual-cylindrical target AC magnetron sputtering source using a computer-automated Langmuir probe system. Electron density, electron temperature, ion density, plasma potential, and floating potential are measured as a function of AC phase and probe position under the large area magnetron source. Measurements are made with a single-tip Langmuir probe mounted to a linear positioning drive. Measurements are gated and delayed from a reference voltage waveform measured between magnetron targets with a time resolution of 1 microsecond. This paper will present the experimental configuration as well as the spatial, and AC phase dependences of these discharge parameters for various gases, process conditions, and target materials.
Due to the increasing demands on size, throughput and precision, the development and optimization process of plasma deposition sources by empirical methods suffers from the increasing complexity and construction costs. For this reason, the development of simulation models for low-pressure plasma deposition processes has been the subject of large R&D efforts during the past decade. However, substantial progress has been possible only within the last few years due to the increasing availability of parallel hard- and software architectures. With the use of massive parallelization we implemented a full featured 3D simulation environment for low pressure gas flow and plasma discharge phenomena based on the Particle-in-Cell Monte-Carlo (PIC-MC) approach, which can be applied on CAD data of industrial coating setups. This work gives an overview of different issues in low-pressure plasma deposition processes, whereof the understanding has been improved by the use of simulation models. For magnetron sputtering, examples are simulation of gas inlet and gas separation systems as well as deposition profile calculations as well as deposition profile calculations by tracing the scattering of sputtered particles in the gas phase. Furthermore, the self-consistent plasma simulation enables the theoretical understanding and circumvention of empirically known plasma anomalies of magnetron devices, which frequently cause non-uniform erosion profiles. For plasma enhanced chemical vapor deposition, the potential of time-dependent RF discharge simulations as well as multi-species transport simulations for predicting the deposition homogeneity as well as the energy bombardment onto the substrate with respect to the plasma source geometry and process parameters are discussed. The low-pressure gas flow and plasma simulation has been proven to be a design tool for efficient development of advanced low-pressure plasma deposition sources. Comments on further possible applications as well as on current issues in theoretical plasma deposition process modeling will be given as an outlook.

Nowadays, the use of rotatable magnetrons has become the standard for high quality thin film deposition in large area architectural and automotive applications. In the PV industry, however, this technology still has a large potential in dealing with current and future needs. Applying the rotatable sputtering technology to PV is a step towards cost-effective mass production, outperforming the capabilities of planar cathodes and even becoming a valuable alternative for some PECVD layers. The success within PV applications however requires fit-for-use sputter hardware as well as targets which are able to live up to the PV standards and future expectations. In this paper, targets and hardware for PV applications will be discussed in more detail. Promising results are discussed on a new concept AZO target, having the potential of surpassing the state-of-the-art properties. High-purity Si targets are presented offering a high quality alternative for PECVD deposited passivation and anti-reflection layers on crystalline Si solar cells. Next to this, other materials may complete the necessary cylindrical target portfolio. Besides the axial magnetron and the compact end-block, which are both fit for use for PV applications; a new adjustable magnet-bar is presented, allowing more accurate tuning and better process control both locally and overall.
Tuesday, May 12

12:10 p.m. L-9 Stress-Free Bonding of Large Linear Sputtering Targets for LCD Displays


Sales of sputtering targets for TFT (thin film transistor)-LCDs are projected to grow at a compound annual growth rate of 30% to reach over a billion dollars by 2012*. Sputtering is used in the manufacturing of aluminum based electrode layers, molybdenum-based wiring and transparent conductive layers (mainly ITO). The glass panel sizes used in the manufacturing of LCD displays have been steadily increasing due to the demand for higher efficiencies and larger displays. The next generation process (Gen 10) will use glass panels measuring 2850 mm x 3050 mm. As the glass panels have expanded in size, so too have the sputtering targets. A Gen 10 sputtering target will measure 3400 mm x 218 mm. Bonding these large targets to CTE (coefficient of thermal expansion) mismatched backing plates is becoming increasingly challenging. RNT has developed the process of bonding stress free, flat bonded assemblies of late generation LCD sputtering targets using reactive multi-layer foil as a localized heat source. This talk will demonstrate the capability of bonding targets to backing plates of sizes up to and including Gen 10 and on material combinations as divergent as molybdenum (CTE = 5.4 x 10^-6/°C) and aluminum (CTE=24x10^-6/°C).

Vendor Innovators Showcase
Tuesday, May 12

10:30 a.m. IS-1 Benefits of Aluminum for the Construction of Vacuum Chambers for Use in Large Area Coating and Deposition Systems

K. Coates, J. Bothell, R. Bothell, and E. Jones, Atlas Technologies, Port Townsend, WA

The mass production of architectural glass, flat panel displays and photovoltaic panels demands ever increasing complexity and scale. Traditionally, high-vacuum coating chambers are constructed from stainless steels such as 304, 304L and 316L. The chambers become a significant component of the system budget. This paper aims to show that aluminum can be considered to have significant advantages over steel, even mild steel, as a material for chamber construction. Over and above total cost (an aluminum chamber is approximately 40% cheaper than an equivalent stainless one), aluminum 6061 alloy has multiple material and vacuum properties that provide strong incentives to produce large vacuum chambers from aluminum. Compared to stainless steel, aluminum is a much easier and quicker material to machine and manufacture from. Its lighter weight allows for easier handling and transportation. Aluminum has a high thermal conductivity that allows for the efficient bakeout of chambers and facilitates the management of process temperatures. Perhaps most importantly, the high vacuum properties of aluminum rival those of stainless steel. Outgassing is exacerbated in large chambers with significant internal surface area. It is possible to achieve outgassing rates that exceed stainless steel, in the region of $1 \times 10^{-13}$ Torr l s$^{-1}$ cm$^{-2}$.

Tuesday, May 12

10:40 a.m. IS-2 Dry Pumps - Cost Effective “Green” Vacuum Coating Process Solutions

J. Luby, Edwards Vacuum, Tewksbury, MA

There is growing pressure on manufacturers to make “green” products, which ties into a responsibility each and every one of us has to make environmentally responsible choices. OEMs and end users alike are faced with fluctuating energy costs having a significant impact on the cost to manufacture and operate a coating tool. Companies which faced the reality of a shifting demand toward “green” products and “green” manufacturing stand ready to supply the world’s demand for high quality products with minimal environmental impact. This paper examines the shift in demand for vacuum products requiring less energy to operate, less oil to dispose of, least footprint to consume, and least cost of ownership while never compromising and perhaps improving performance and longevity. From conception to prototype to test to market, one must look for ways to make a better product for less money and less environmental impact. For many companies, this effort began long before being “green” was fashionable or even considered a phrase to be coined. Being “green” is becoming the rule and we must look at what it takes to manufacture a product but, perhaps more importantly, we must better understand the cost of the product over its lifetime to know if we are striving to be “green”.

10:50 a.m. IS-3 Design Considerations of Precision Planetary Rotation Systems

J. Oliver, Vacuum Innovations, LLC, Pittsford, NY

Planetary rotation systems have one fundamental purpose, to improve thin-film uniformity and thickness control. The Vacuum Innovations planetary incorporates advanced design and performance features while maintaining a focus on reliability and ease of maintenance. The resulting design requirements are far-reaching, from careful tolerancing of flatness and height, integrated mounts for fixed-position uniformity masks, and integrated multi-point crystal monitoring. We carefully characterize the impact of different gear ratios, source locations, and planet sizes. By modeling the influence of each of these characteristics on the overall performance of the system, we are able to achieve far more stable control and uniform coatings. We provide uniformity masks customized to individual system geometry for optimal performance and refine the designs based on a given deposition process, to compensate for the influence of gas partial pressures, source materials, and electron-beam sweep patterns. Even large-diameter substrates can be coated with less than 0.25% film non-uniformity.

11:00 a.m. IS-4 Successful Application of Varian, Inc.’s Unique Remote AssistTM Program for Large Turbomolecular Pumps Used in Vacuum Coating Systems

T.C. Forbes, Varian, Inc., Lexington, MA

Varian, Inc. has successfully deployed its latest innovation in customer service products on vacuum coaters worldwide: the Remote Assist customer support program. Unique in the industry, Remote AssistTM is a proactive program that is specifically designed to provide first-class support in monitoring and servicing Varian turbo pumps by combining Varian’s remote Monitorr diagnostic system, extended warranty, and Varian, Inc’s advanced exchange program. This allows for maximum up-time and system availability. This Innovators Showcase Power Point presentation will describe the Remote Assist program and its implementation on coaters using Varian’s large turbo pumps around the world.
High Power Impulse Magnetron Sputtering (HIPIMS) offers the ability to apply new and already existing thin film materials with advanced film properties, on standard coating machines. To deposit these state-of-the-art thin films to a structured surface, a functional substrate bias voltage system is a basic necessity. Due to the high density plasma and the high ionization rate, it is a “must” that the applied bias voltage is present on the substrate during the short HIPIMS-impulses, when peak power densities of kW/cm$^2$ are present. It can be experienced with conventional bias power supplies, that the bias voltage drops dramatically (or even vanishes) during the few microsecond lasting HIPIMS-impulses. This is well known. But there seems to be a lack of industrial available and reliable power supplies to handle this condition. Based on its already proven Magix bias PS technology with the “patented tapless wide output load impedance range”, Solvix has developed such a bias-power-system with a highly sophisticated arc-management. This new Solvix DC and DC-pulse HIPIMS bias-supply can be applicable for all the conventional techniques such as a magnetron-sputtering, arc-evaporation, pre-clean and ion-etching. Plus, it has the capability of handling all the requirements needed for the HIPIMS-impulses.

In situ optical monitoring is rapidly becoming a crucial technology in the manufacturing of complex high precision optical coatings. Recent hardware and software based technological advances in optical monitoring will be presented. The first of these is real-time refractive index compensation during the coating process. This is especially useful when depositing TiO$_2$ layers which can experience significant run-to-run refractive index variation due to the different oxide states achievable. Achieving a fast and accurate measurement of the actual refractive index during the coating run, and then accurately correcting the cut points for all of the subsequent layers leads to enhanced process yield and product performance. Other developments include extension of monitoring wavelength regimes down into the UV and out into the IR, new test glass changer configurations for different chamber geometries and new remote support and monitoring capabilities.
Tuesday, May 12

11:30 a.m. IS-7 Current and Future Development of Arbitrary Voltage Waveform Pulsed Plasma Generators
R. Chisyakov and B. Abraham, Zond Inc./Zpulser, LLC, Mansfield, MA

A unipolar arbitrary voltage wave form pulsed plasma generator (Zpulser) was introduced as a new development in pulsed DC technology for material processing applications. When used for magnetron sputtering applications, the Zpulser output voltage pulse shape typically has two voltage stages that create both low power and high power magnetron discharges within a single pulse. The high power magnetron discharge stage generates a high intensity flux of ionized sputtered target material atoms. By adjusting voltage rise time and voltage oscillation amplitude within single pulse, a stable high density plasma discharge with low energy metal ions at a high deposition rate can be created for both reactive and non-reactive processes. Zpulsers can also be used for cathodic arc deposition to improve the ionization level and control the size of macro particles. The principles of operation of the Zpulser as well as the method of programming an arbitrary voltage pulse shape will be discussed. Integration of Zpulsers into PVD tools for industrial applications will also be discussed. A road map for integrating Zpulser plasma generators into PVD, CVD and RIE applications will be presented.

Tuesday, May 12

11:40 a.m. IS-8 COPRA 300-Radio Frequency Plasma Source for Enhancement to Existing PVD Coaters
W. Schwärzler and M. Kessler, Provac AG, Balzers, Principality of Liechtenstein; and D. Gary, Ricmar Sales and Service Inc., Aptos, CA

Provac AG introduces the industry’s first stand alone RF Plasma Source enabling vacuum coaters to easily upgrade their existing PVD systems (chambers from 650 mm/25” to 1100 mm/43”). Based on the well established and proven Taurion Series RF-PEPVD technology, the COPRA 300 consists of an RF Plasma Source and portable workstation module containing laptop PC, control system, mass flow controller, power generator, and connection lines. By upgrading your existing coater or replacing the current ion source, the COPRA 300 enables the production of densified oxide layers (SiO2, TiO2, Ta2O5, Nb2O5, and more), shift free layers (filters), and nitrate layers. Additionally, you can plasma etch, cleanse and degas substrate surfaces in a free environment. The COPRA 300 is very simple to integrate and operate, 400+ hours of operation without maintenance; consisting of a cloth wipe down and possible extraction grid replacement, enables use of the basically neutral plasma generated from the high-frequency plasma source eliminating potential damage to the substrates by electrical discharges, and provides very high long term stability. Additionally, the use of pure process gasses (i.e. oxygen and argon) expands new areas of applications, where other processes are not suitable.
Magnetron sputter cathodes, with rotating tubes as the target, eliminate several of the problems associated with planar magnetrons. However, there is still a need to optimize performance via the magnetic circuit that confines the plasma. A range of different magnetic types will be illustrated that can improve film uniformity, TCO conductivity, plasma stability as well as the deposition rate and energy consumption.
Tuesday Afternoon
May 12, 2009
Tribological and Decorative Coating
Sn thin films show high electrical resistance at critical thickness and typical bright metallic color. The growth mode of Sn films is island mode so that, at critical thickness, the islands of Sn do not connect with each other. This is the reason of the high electric resistance of Sn films. Therefore, the Sn thin film is a candidate for metallic coating of mobile information devices. For the deposition of Sn films, thermal evaporation and magnetron sputtering systems were used. The Sn films deposited with thermal evaporation showed the regular island size and easy control of island size with amount of Sn source. However, the films deposited with magnetron sputtering showed relatively small island size, and the island size did not control by process conditions. The small island size of Sn films deposited with sputtering affected the color of the film, which was relatively dark metallic color compared with that of the films deposited with evaporation. The adhesion of Sn films deposited with sputtering was better than that of the films deposited with evaporation.

The current state of the art for the coloured coatings is based on the interference of the light reflecting from the metal/metal oxide interface. This work addresses coloured coatings based on the surface plasmon resonance (SPRs)-induced absorption in a specific wavelength range. In this case, hybrid materials consisting of metal nanoparticles dispersed in a dielectric matrix are the subject of interest due to their functional properties for ultra thin colour filters and decorative coloured coatings. Inorganic films are particularly attractive as matrix for hard and wear-resistant coatings. Various approaches have been reported to incorporate metal nanoparticles into the host dielectric medium. The present work is concerned with the preparation of inorganic/metal nanocomposites by means of a hybrid technique, Plasma-Enhanced CVD and magnetron sputtering for the deposition of the dielectric medium and the metallic clusters, respectively. The optical properties of the nano-composite (NC) layers have been investigated by UV-VIS-NIR variable angle spectroscopic ellipsometry (VASE) and by optical transmission measurements. Rutherford backscattering/ Elastic Recoil Detection (RBS/ ERD) and InfraRed absorption spectroscopy (FTIR) were used for characterizing the film density, chemical composition and environment. AFM and TEM observation as well as the EDX analysis revealed the formation of nanoclusters.
The activity aiming to replace galvanic treatments by vacuum processes is based on the effort to reduce the production costs and to set-up new decorative effects impossible to be galvanically realised because the use of dielectric materials. This work describes cost effective metal-oxide and oxide-metal-oxide decorative coatings based on the use of titanium as basic material deposited by Ion Plating Plasma Assisted from Reactive Magnetron Sputtering source. A new multilayer protective and decorative structure, based on the use of titanium and titanium oxides is also presented. The protective effect against aggressive environment is obtained by alternating layers of metal and metal-oxide mixture that exhibit good barrier properties because its microstructure. The decorative effect is obtained with a final couple of titanium and titanium dioxide or, on transparent substrates, with a titanium dioxide-titanium-titanium dioxide structure providing different colour effects on the two faces. Cost effectiveness is obtained by reducing dramatically the process requirements and duration, by simplifying the preparation and cleaning phase, reducing the pump-down time and depositing at room temperature. The treatments were realised and characterised on aluminium, stainless steel and glass substrates.

ZrN and ZrCN are widely applied coatings systems, for industrial components and decorative applications. ZrON has some interesting properties linked with low surface energies. HIPIMS has been used to deposit nitrides and ternary alloys of ZrN, ZrON and ZrCN. For having a systematic comparison we deposited and characterized ZrN produced by ARC, UBM and HIPIMS technologies. The diversity found in terms of structure, mechanical properties, surface energy, corrosion resistance or simply in the colour could be still further varied by the addition of third elements such as oxygen and carbon. HIPIMS-ZrN evidenced dense structure as well as improved corrosion properties compared to other coating technologies. If on the one hand the addition of carbon in HIPIMS-Zr(C)N could allow us to obtain black colour with improved wear behaviour, the addition of oxygen to HIPIMS-ZrN was done to attempt producing combinations of antibacterial and transparent coatings. The structure of the coatings has been studied by XRD and observed by cross sectional SEM. Mechanical properties like coating hardness and elastic modulus were determined by nanoindentation techniques.
Tuesday, May 12

2:50 p.m. T-11 Decorative Coatings Obtained by Combination of PVD, Galvanic and Powder Coatings


PVD is a powerful technology for the development of decorative coatings offering interesting properties: bright metallic appearance for polymer substrates and a wide range of colors. But at the same time, this technique presents some limitations like insufficient leveling and covering of substrate defects and poor corrosion protection. The combination of the PVD processing with other coating techniques enables efficient use of the particular advantages of each coating technique. In the present work three different combinations have been investigated: 1) PVD start layer as a base for an electroplated finishing, 2) powder coating as base for decorative PVD and 3) PVD on nickel electroplated substrate. Combination of magnetron sputtering and arc evaporation was used in order to take advantages from both processes. In the first case, it has been seen that the PVD start layer replaces at least four pre-treatment steps needed for full electroplating. Even more, PVD start layer could be used to make a conductive layer on plastics that cannot be usually electroplated (like polypropylene or polyamide which have increasing interest due to economical aspects). The second combination, a first powder coating, leads to products with good surface finishing avoiding the polishing step, an improved adhesion of the PVD coating and a good corrosion protection of the substrate. The deposition of a PVD alumina layer on top of the epoxy powder coating before depositing the PVD film significantly improves adhesion, chemical and heat resistance of the coating system. Finally, it was observed that adhesion of PVD coating on electroplated nickel depends highly on the characteristics of the base coating.
Cleantech Symposium
Although the modern solar industry has its roots in North America, in recent years, the focus of market development efforts and capital investment has been in Asia and Europe. While these two markets are unquestionably the largest, the market growth rate in North America has accelerated in recent years. North America is a smaller but more attractive market because its forecasted growth shows no sign of abating. The prospective growth is not confined to any particular segment. C-Si, thin film and CSP (concentrating solar power) all have strong growth momentum and the ability to attract investment capital to fund the growth. The underlying technologies in each of these segments have different drivers that will influence adoption and commercialization rates. This presentation will examine each of these segments and discuss growth, challenges and opportunities going forward. As of this writing, it appears that an Energy Bill will be passed and signed into law; this too will play an important role in developing the market going forward.

For thin film photovoltaic applications, functional layers are inevitable in terms of electrical insulation and diffusion resistance. The change of substrate material from rigid glass to flexible metal foil for thin film solar cell production demands the prevention of diffusion into the absorber layer of impurity atoms out of the metal. Furthermore, modules with monolithic serial connection need an electrical separation of the cell back contact from the conducting metal substrate. SiO$_x$-based layers on steel strips were produced by electron beam (EB) technology, hollow cathode arc activated deposition (HAD-process), plasma polymerization and by combining these three processes. These methods are distinguished by their in-line capability enabling roll-to-roll processing with dynamic rates of up to 1 $\mu$m$^3$/s. Moreover, the possibility arises to produce coatings scaled up to common industrial width and larger dimensions. Electrical and dielectrical examinations of the layers were carried out to characterize the insulating abilities. Also microstructure and layer growth were analyzed. The efficiency of the barrier properties was determined by SIMS-measurements. The insulating properties are distinguished by the breakdown voltage of some hundred volts. The positive effect of the barrier layer is apparent from the strong reduction of Fe diffusion into the CIGS-layer.
Tuesday, May 12

2:30 p.m. CT-2 PECVD Thin Films for Anti-Reflection and Passivation of Crystalline Silicon Solar Cells

M. George, H. Chandra, and J.E. Madocks, General Plasma, Inc., Tucson, AZ

Crystalline silicon solar cells owe their high efficiencies in part to advanced high throughput thin film technologies. These thin films serve both as optically matched anti-reflective layers and simultaneously render the surfaces of the underlying active semiconductor passive. Hydrogenated silicon nitride is of keen interest because it is a key thin film for commercial crystalline silicon n⁺ emitter solar photovoltaic devices. A new Plasma Enhanced Chemical Vapor process has developed that deposits thin films for this demanding requirement: an advanced hydrogenated SiN:H process capable of depositing SiN:H films on n-type FZ Si surfaces with minority carrier lifetimes exceeding 2 milliseconds, measured by photocconductivity decay. Deposition conditions include temperatures between 200°C and 350°C and pressures from 20 to 75 millitorr - yielding a SiH₄ utilization efficiency that exceeds 10% at deposition rates >60 nm-m/min. We discuss the thermal stability, thin film bonding and surface chemistry of the SiN:H films that account for this exceptional surface passivation. Future crystalline silicon cells will be manufactured with p⁺ emitters, enabling efficiency improvements of 2-5% over 21% efficient n⁺ emitter cell designs. This cell architecture requires films with altogether different properties from SiN:H to enable these dramatic efficiency gains. We include summary data on thin films deposited by our Dual PBS, PECVD source for this application that include index of refraction, minority carrier lifetime and deposition rates.

Tuesday, May 12

2:50 p.m. CT-3 Stochastic Nanostructures on Polymers for Solar Applications

N. Kaiser, K. Fuechsel, U. Blumrueder, P. Munzert, and U. Schulz, Fraunhofer Institute for Applied Optics and Precision Engineering IOF, Jena, Germany

A new technology based on plasma etching has been developed to produce antireflective nanostructures. By choosing thin initial layers and variable plasma conditions a broad range of differently shaped structures can be produced on various polymers. A broadband antireflective effect can be achieved that is less sensitive to the incident angle of light compared to multilayer interference coatings. In combination with metallic layers, some types of structured surfaces can be transformed to show high absorption. Such surfaces appear completely black in the visible spectral range. This paper presents structured polymer surfaces exhibiting broad band antireflective properties optimized for the solar wavelength range as well as absorbing surfaces produced by plasma etching.
Recently, there has been considerable activity scaling thin film photovoltaics from R&D to high volume manufacturing because of their low cost potential. For CIGS thin film solar, the manufacturing challenges focus on optimizing the deposition for high material utilization, low thickness variation and decreased system footprint. In this contribution we report on a deposition modeling and optimization method that evaluates parameters such as number of sources, individual flux rates, source-to-substrate distance, source angle, source separation and deposition area in order to quantify the impact of these variables and their dependency on one another. We have used this process for a variety of deposition configurations including thermal point sources for copper, indium, gallium and an array of nozzles for selenium linear sources integrated into web and inline glass systems. The modeling process provides a fast, cost effective and low risk means of determining the optimal system design given the process and hardware constraints. This method is applicable for substrate sizes beyond 1m and we will present system configuration results for substrate sizes ranging from 300mm to 1200mm for copper, indium, gallium and selenium sources illustrating that material utilizations up to 75% can be obtained with thickness variations +/- 5%.
Live Product Demonstrations
Tuesday, May 12

12:10 p.m. LP-1 A Demonstration of Theory and Methods for Cost-Effective Nanoindentation

N. Randall and E. Skopinski, CSM Instruments, Needham, MA

Instrumented nanoindentation is a widely used method for the analysis of thin films and coating systems. In this live demonstration, an exemplary nanoindentation study will be performed. Focus will be given to the relation of results to basic theory, to provide understanding on the theoretical source and meaning of instrumented indentation results. The CSM Table Top Instrument combines all the advantages of the Standard Nanoindentation Tester into a small and simple-to-use instrument which is ideally suited to routine nanoindentation testing where a Full Platform System may not be appropriate. The nanoindentation head offers the same high performance as the standard Nanoindentation Tester (NHT) head. It is also upgradeable with optional technical features like the Sinus mode (Dynamic Mechanical Analysis: storage and loss moduli) and can be configured with a range of displacement stage options and integrated video microscope. The Load Range is 0.1 to 500 mN, the Load Resolution is 0.04 µN, the Max Depth is 200 µm and the Depth Resolution is 0.04 nm. CSM Table Top Nano Indentation Tester is a cost-effective solution which meets the ISO 14577 & ASTM E2546 standards.

- Easy to use
- Full NanoIndentation Functionality
- Robust Head
- Compact Frame
- Manual or Motorized Table

Tuesday, May 12

12:45 p.m. LP-2 Demonstration of Sputter Target Bonding at Room Temperature Using NanoFoil®

M. O’Neill and A. Duckham, Reactive NanoTechnologies, Inc., Hunt Valley, MD

NanoBond® is a process that enables solder bonding of materials in nanoseconds. This is accomplished by inserting a sheet of NanoFoil® in between two surfaces prepared with prewet solder. Under pressure, the NanoFoil® is ignited and serves as an embedded, sacrificial heat source. The soldered assembly is bonded with little evidence of deflection or stress created by the bonding process. The NanoBond® process has been adopted by several companies who bond sputter targets to a backing plate. NanoBond® is used to bond metal and ceramic oxide targets to a range of backing plate materials. In many cases this combination possess a temperature coefficient of expansion mismatch (TCE). The NanoBond® process only heats the interface being bonded, avoiding any stress or deflection due to the mismatch, creating a strong and reliable bond. Due to the flexibility in using any solder, the NanoBond® process enables higher power sputtering, reduction or elimination of arcing, use of lighter and less expensive backing plates, and an overall increase in sputter tool uptime. The NanoBond®process can be conducted in stages, allowing for improved manufacturing flow and enabling the ability to bond sputter targets live for the participants of SVC 2009.
Tuesday, May 12

1:20 p.m. LP-3 Better Utilization with Cylindrical Magnetron

R. Newcomb and J. Hrebik, Angstrom Sciences Inc., Duquesne, PA

Angstrom Sciences has created a compact, lightweight, and economical cylindrical magnetron specifically designed to offer uniform thin films while maximizing target utilization. Cylindrical Magnetrons can hold up to 3 times the amount of target material as a planar magnetron with the same spatial area. The ONYX-Revolution further optimizes material investment with greater than 85% target utilization and with a universal and cost-effective magnet design, is able to fit any target type.
Vendor Innovators Showcase
Tuesday, May 12

2:00 p.m. IS-10 Advanced Sputter Magnetrons and Linear Ion Sources
J. Madocks, General Plasma, Inc., Tucson, AZ

With the acquisition of Advanced Energy’s linear ion source product line in 2008, General Plasma is the world leader in high performance ion sources for solar, glass and web applications. In a whirlwind tour, attendees are shown the latest in ion source performance statistics and application examples. This is a quick overview to leave time for presentation of General Plasma’s new Compact End Block for rotary magnetron sputtering. The innovative end block has mechanical water seals, ferrofluid vacuum seals and is only 100mm long! Any time left will be used to share the latest user data from our Moving Magnet Planar Magnetron. Exciting stuff!

Tuesday, May 12

2:10 p.m. IS-11 Controling Utilization and Uniformity
R. Newcomb, Angstrom Sciences, Inc., Duquesne, PA

Angstrom Sciences has developed a magnetron with an electromagnetic coil that enables you to Control the Plasma across the surface of the target.
Tuesday, May 12  
2:20 p.m. IS-12 Technology Update: NanoBonding of Sputter Targets  
M. O’Neill and A. Duckham, Reactive NanoTechnologies, Inc., Hunt Valley, MD  

NanoBond® is a process that enables solder bonding of materials in nanoseconds. This is accomplished by inserting a sheet of NanoFoil® in between two surfaces prepared with prewet solder. Under pressure, the NanoFoil® is ignited and serves as an embedded, sacrificial heat source. The soldered assembly is bonded with no evidence of deflection or stress created by the bonding process. The NanoBond® process has been adopted by several companies who bond sputter targets to a backing plate. NanoBond® is used to bond metal and ceramic oxide targets to a range of backing plate materials. In many cases these combination possess a temperature coefficient of expansion mismatch (TCE). The NanoBond® process only heats the interface being bonded, avoiding any stress or deflection due to the mismatch, creating a strong and reliable bond. Due to the flexibility in using any solder, the NanoBond® process enables higher power sputtering, reduction or elimination of arcing, use of lighter and less expensive backing plates, and an overall increase in sputter tool uptime. Several recent case studies of successful sputter target bonding will be presented.

Tuesday, May 12  
2:30 p.m. IS-13 Deposition Tolerant Langmuir Probe  
D. Gahan, National Centre for Plasma Science and Technology, Dublin, Ireland; and B. Dolinaj and M. B. Hopkins, Impedans Ltd., Dublin, Ireland  

In a standard Langmuir probe system, the monitoring of plasma parameters during plasma deposition is limited by the effects of probe surface contamination. A number of issues arise: i. A Langmuir probe immersed in the plasma during deposition processes is subjected to the deposition of a layer resulting in a large disturbance of the probe characteristic. Surface contamination changes the work function, resulting in a shift of the probe characteristic and/or in hysteresis in the I-V Characteristic. The formation of dielectric layers causes the slope of the characteristic to become shallow and eventually reduce the current to zero. This problem is addressed in current Langmuir probe systems based on electron or ion cleaning but limits the probe operation to plasma with low deposition rates. ii. A poor ground return path for the electron current causes shifts in the plasma potential. While this problem is addressed in modern probe systems by using a floating reference probe to compensate for low frequency effects, in deposition plasmas the reference electrode cannot be cleaned by electron bombardment and may be become insulating. The poor electron ground return is made worse by insulating coatings on the wall. In order to produce a Langmuir probe that can operate well in deposition plasma we have introduced a high frequency swept probe. The probe attains a DC bias negative relative to the plasma potential and draws a net current close to zero. The probe records the AC IV characteristic or complex impedance of the sheath and determines the plasma parameters. This technique is valid even in the case of a fully insulating layer forming on the probe surface. The probe draws little net current and minimal ground return is required. We show that the plasma to ground sheath capacitance provides sufficient current during the electron collection period. A unique feature of the probe is the ability to attain a bias voltage above the plasma potential even when coated with a non-conducting layer. We show results of the system in an O₂/N₂ plasma and compare the swept probe with a standard Langmuir probe.
Tuesday, May 12

2:40 p.m. IS-14 Investigation of Creep Behaviour with a New Innovative Nanoindentation Tester

N. Conte and R. Consiglio, CSM Industries SA, Peseux, Switzerland; and N. Randall and E. Skopinski, CSM Instruments Inc., Needham, MA

Nanoindentation testing is particularly appropriate for creep and stress relaxation tests because it can measure materials whose properties are highly viscoelastic. However, the main drawback of nanoindentation tests is linked to the low thermal stability of most instruments. These instabilities introduce an uncontrollable penetration drift superimposed to the viscoelastic deformation of the sample. For some very thin coatings, thermal expansion of the instrument frame can cause significant measurement error. The recent development of a new innovative instrument (the Ultra Nanoindentation Tester) has allowed such drawbacks to be avoided, and has allowed the precise investigation of the creep behaviour of samples using very long duration tests. These results were made possible thanks to a quasi elimination of the thermal drift by the use of specific dedicated materials with very low thermal expansion coefficients and a special design of the measurement head. Furthermore, the influence of the deformation of the frame has been eliminated due to an active top referencing which continuously monitors the position of the surface of the sample through a reference applying a very small and controlled pressure. A series of long time quasi static tests various coatings and will be used to demonstrate the efficiency of this novel instrument design and ability.

Tuesday, May 12

2:50 p.m. IS-15 Surface Modification by 3D Nano Coatings

M. Fliedner, Cotec GmbH, Karlstein, Germany

Easy-to-clean coatings are a special kind of protection coating, only a few nanometers thick, against mechanical stress, watermarks, fingerprints, dust, grease and so on. The combination of coating systems and an ultra hydrophobic material will leave a long lasting coating. The hydrophobic and oleophobic performance will remain the entire lifetime of the product. New test equipment and background data on the physical basis of these properties are presented. Surface characterization was performed by electron microscopy and detailed contact angle measurements.
Plasma Processing
8:30 a.m. P-4 Plasma-Based Approach to Controlling the Oxygen Concentration in Graphene Oxide

S.G. Walton, E.H. Lock, and M. Baraket, Plasma Physics Division, U.S. Naval Research Laboratory, Washington, DC; P.E. Sheehan and Z. Wei, Chemistry Division, U.S. Naval Research Laboratory, Washington, DC; and J.T. Robinson and E.S. Snow, Electronics Division, U.S. Naval Research Laboratories, Washington, DC

Graphene and graphene oxide have attracted widespread interest because of their unique structural and electronic properties, which lend themselves well to the production of nanoscale electronic and sensing devices. Indeed, graphene is a single monolayer thick carbon sheet with remarkably high electron mobility. The addition of oxygen increases the sheet thickness and decreases this mobility and, more interestingly, determines both the conducting and reactive properties of the material. One significant impediment to realizing the potential of graphene oxide is the development of an industrially viable approach to producing large area substrates of well-controlled oxygen concentration. In this respect, plasmas are an ideal candidate but problems associated with the large fluxes of energetic ions are a significant concern. The Naval Research Laboratory has developed a plasma processing system that is characterized by a uniquely low incident ion energy, in the range of a few eV, at or near the carbon-carbon and carbon-oxygen bond strength. In this talk, we discuss the use of this system to reduce the oxygen concentration of graphene oxide. We examine both the processing conditions and the resulting properties of the material. This work was supported by the Office of Naval Research.

8:50 a.m. P-5 Modeling the Plasma Growth of Carbon Nanostructured Materials

E. Neyts, M. Eckert, A. Maeyens, and A. Bogaerts, University of Antwerp, Antwerp, Belgium

Invited 40 min. Talk

Carbon nanostructured materials such as carbon nanotubes (CNTs), graphene, amorphous carbon films, and (ultra) nanocrystalline diamond, attract a lot of attention thanks to their exceptional electronic, optical, mechanical, and tribological properties. Each of these materials can be deposited using plasma sources starting from some hydrocarbon gas, and hence it is of great importance to obtain a fundamental understanding of the plasma used to deposit these materials. For this purpose, we use self-consistent fluid simulations. In the first part of the presentation, attention will be focused on how different operating conditions lead to a different plasma chemistry and plasma composition. Beside a computer model for the plasma itself, it is also of great interest to simulate the interaction between the plasma and the walls of the plasma reactor, because (i) this defines the boundary conditions of the plasma simulations, and (ii) it provides information on the growth mechanisms of the deposited materials. For this purpose, we apply molecular dynamics (MD) simulations. In the second part of the presentation, the capabilities and limitations of MD simulations will be illustrated for the case of deposition of various kinds of carbon nanostructured materials, including amorphous hydrogenated carbon thin films, (ultra)nanocrystalline diamond and carbon nanotubes.
SiO$_2$ layers have been deposited in a remote expanding thermal plasma setup enabling a good control of the ion flux (by changing the deposition chemistry and the arc plasma parameters) and the ion energy by an additional RF substrate biasing or a tailored ion biasing technique. The role of ion energy and ion-to-growth flux ratio on the film densification has been investigated by means of ellipsometric porosimetry, monitoring the refractive index change due to the adsorption (and desorption) of ethanol vapors in the volume of macro-meso-micro pores. From the analysis of the adsorption isotherm and the presence of hysteresis, the open porosity in the layer can be determined. Both biasing techniques lead to densification of the deposited layer, which experiences a transition from mesoporosity to microporosity and eventually non-porosity. Although both biasing techniques also lead to a comparable critical ion energy value per deposited SiO$_2$ unit, the ion-to-growth flux ratio and ion energy are not found to be interchangeable. While the RF biasing technique allows the above-mentioned transition in a relatively small bias voltage range (down to 50V), the layers densified via the tailored ion biasing technique require a larger voltage range (down to 240V) to quantitatively remove microporosity.

Plasma modification of powder has recently attracted much interest because of new perspectives of the interfacial properties supervision. Plasma modified powder was used as a filler for composite materials or for production of parts using roto-molding technique. Parts sintered from the plasma modified polyethylene powder preserved high surface tension, which allowed e.g. direct painting or adhesive bonding without any additional pretreatment or using them as a filler in composite materials. Plasma modification also significantly enhanced adhesion of the polymer to the substrate. Various methods for powder treatment have been reported for low-pressure plasma treatment of powder including fluidized bed or reactors with mechanical stirring. Based on laboratory experiments and theoretical analysis of various treatment methods a concept for industrial scale production of plasma-modified powder was proposed. The principle of the industrial-scale reactor will be presented. Production capacity and cost estimation will be also presented. Partial support of the projects MSM 4674788501 and GACR 106/08/1665 is greatly acknowledged.
Wednesday, May 13

10:30 a.m. P-8 Detecting and Preventing Instabilities in Plasma Processes


RF driven plasmas commonly used in enhanced CVD deposition and etch processes are continually subject to shrinking feature size and film thickness requirements. The ongoing evolution of film architectures has driven many processes into regimes not previously explored with past technologies. Low pressure, low power processes often using electronegative gases pose unique challenges to power delivery in these increasingly demanding applications. All of these directly influence plasma impedance, a critical characteristic of any RF driven plasma. Besides the need to maintain load impedance in a range for efficient power delivery, the dynamic sensitivity of plasma impedance to other process variables can prove critical to accurate power delivery into plasma processes. Low pressure, low power, electronegative plasmas are particularly prone to stability issues, some inherent to the plasma and others resulting from dynamic interaction between the plasma impedance and the power delivery system. Today’s RF power systems, in addition to becoming increasingly efficient, are also equipped with state-of-the-art measurement, regulation and control features. When properly implemented, advanced features such as variable frequency, delivered power regulation and high speed impedance measurement can be used to not only detect the presence of plasma instabilities but also to suppress and avoid them. In this paper we demonstrate the use of advanced power delivery and measurement technologies for the detection and prevention of plasma instabilities. We show characterization methods that can be used for determining process margin and effective means for power delivery optimization for ensuring process stability. For the most sensitive processes, we further demonstrate an implementation of a real time control system for plasma stabilization that detects the presence of instabilities and actively corrects them to restore stable power delivery and plasma conditions.

Wednesday, May 13

10:50 a.m. P-9 Pulsed Heating for Plasma Assisted Processes, Principles and Application

E. Bergmann and A. Houbbi, University of Applied Science of Western Switzerland, Geneva, Switzerland; and B. Pecz and L. Toth, Műszaki Fizikai és Anyagtudományi Kutatóintézet, Budapest, Hungary

Pulsed heating in plasma assisted deposition processes is a new concept that uses the decay of propagating heat pulses to grow coatings at temperatures significantly higher than the substrate temperatures. Although different options exist for heat pulses and lasers have been used in the past for superconductive oxide coatings, electron bombardment is the method of choice in plasma assisted processes. In practice duration and frequency of the pulses will be linked to the coating growth rate. This allows the deposition of fully tempered coatings or the deposition of laminar structures consisting of sequences of tempered and untempered layers. The validity of the concept is shown by direct observation of the heat wave in a model experiment, from which the surface temperature reached during heating can be estimated. The experiments were carried out in a sputter configuration and the maximum surface temperatures exceed 1000° C. One example of an application are CrC/a-C:H coatings, where pulsed heating leads to higher graphitisatation and a lower friction coefficient. Another example are arc co-evaporated CrC coatings, where pulsed heating can be used to improve the crystallinity of the chromium carbide. In this case the tempered layers appear as galaxies in dark field transmission microscope cross sections. The concept can be used with any plasma assisted vapour deposition technology, but arc evaporation is particularly suited because of the high heating currents, that can be achieved. In industrial systems, coating temperatures exceeding the substrate temperature by 150° C - 300° C can be achieved.
Wednesday, May 13

11:10 a.m. P-10 Recent Advances in APP-Based Food and Non-Food Decontamination/Sterilization

J. Engemann, JE PlasmaConsult GmbH, Wuppertal, Germany (Presented by T. Engemann, JE Plasma Consult GmbH, Wuppertal, Germany)

A technology has been developed recently to generate an extremely low-cost atmospheric pressure dielectric barrier discharge. The necessary electrodes can be printed on disposable labels which in turn are attached to airtight flexible or rigid packages containing the goods to be decontaminated or sterilized. When generating a plasma, these labels need to be only of a limited lifetime in the order of several 10sec to several minutes. During that time, ozone is generated very efficiently with ozone concentration increases of up to more than 200 ppm/sec. Maximum concentrations achieved so far in a suitable gas atmosphere are in the order of 40,000ppm. For comparison, inactivating ozone concentrations for bac. Escherichia, bac. Streptococcus, bac. Clostridium Botulinum and bac. Staphylococcus Aureus range from 200 to about 1000 ppm. Plasma label yields these concentrations after 4 to 5 sec. When switching-off the power ozone decomposes again to oxygen within minutes. This means that upon opening the packages after a suitable time, no harmful ozone is being released. Depending on the surface textures of food (e.g. fruit, meat) an initial microbial load of $10^6$ was reduced by log 3, log 2 and log 1 for apples, chicken- and pork-meat, respectively. More importantly, the subsequent increase of the surviving microorganism population after opening the package was slowed down considerably compared to untreated samples. Textiles for clinical use also need to be sterilized preferentially on the point of use. Here the discharge effectively sterilized the textiles consistently with a reduction of log 6.

Wednesday, May 13

11:30 a.m. P-11 Plasma Enhanced PVD Coatings for 120mm and 155mm Barrel Applications

S.L. Lee, U.S. Army ARDEC-Benét Laboratories, Watervliet, NY; R. Wei, Southwest Research Institute, San Antonio, TX; M. Todaro, U.S. Army ARDEC-Benét Laboratories, Watervliet, NY; E. Langa, Southwest Research Institute, San Antonio, TX; and S. Smith, U.S. Army ARDEC-Benét Laboratories, Watervliet, NY

Plasma enhanced PVD (physical vapor deposition) process with externally generated plasma and substrate biasing were used to deposit environmental friendly protective coatings for potential barrel applications. In this work, tantalum coatings of 200-500µm thickness were sputter deposited on ASTM A723 steel samples cut from curved 120mm smooth bore, and rifled 155mm gun bore sections. SEM showed dense, uniform, adhesive, and crack-free coatings on all bore surfaces, except the walls of rifled 155mm, expected due to oblique angle deposition. XRD showed body-centered-cubic tantalum coatings. Fracture tantalum surface showed excellent microvoid coalescence with ductile mode of fracture, resilient to thermal shock cracking. Adhesion tests performed included groove test, cyclic pulsed laser heating test, and vented erosion simulator test, which simulates the thermal-mechanical-chemical environment of large cal. The 120mm sample showed excellent adhesion, no delamination, no cracks after 129 rounds firing simulated high erosive rounds. The 155mm land and groove surfaces showed excellent adhesion, no delamination, no cracks after 10 cycles of very aggressive pulsed laser heating testing. The plasma enhanced PVD deposition process and the analytic and adhesion test results will be discussed.
Tribological and Decorative Coating
Wednesday, May 13

8:30 a.m. T-6 Present Status of Carbon Based Cutting Tool Coatings for Soft Alloy Workpiece Materials

G.J. van der Kolk, Ionbond Netherlands, Venlo, The Netherlands; and E. Damond, Ionbond France, Chassieu, France

Cutting tool coatings are increasingly optimized for the dominant wear phenomena. For soft metals and alloys like Al-alloys and Ti-alloys adhesive wear is one of the dominant wear mechanisms. Coatings provided for these applications are focusing partly on abrasive wear reduction by having a high hardness, partly on avoiding adhesive wear by working on the surface energies, and avoiding matching with the workpiece materials. The applied coatings range from pure diamond, diamond like carbon (DLC) in many variations, to nitrides and carbo-nitrides. An overview will be presented of the present state-of-the-art coatings. Further, a model will be discussed trying to link the coating engineering to the expected wear mechanisms due to the cutting conditions and workpiece material. Special emphasis will be on multilayer coatings produced by unbalanced magnetron technology, mainly based on a load carrying layer of CrN with a top coating of hydrogenated DLC (CrN/a-C:H) and on non-hydrogenated DLC coatings produced by ARC technology (ta-C).

Wednesday, May 13

9:10 a.m. T-13 DLC Base Coating on Plastics as Support for Scratch Resistant Decorative Finishes


PVD colour coatings are widely spread since the early 1990s due to their high hardness in comparison with galvanic coatings, thus protecting the colour for much longer periods of time than ever achievable with galvanic coatings or lacquers. Because decorative PVD coatings are relatively thin (0.5 micron), their high hardness is only advantageous when deposited on a adequate support layer. For plastics this support is often provided for by a galvanic coating, while for stainless steels it is provided for by a thicker PVD base layer. DLCs can be used as an alternative, since they also exhibit high hardness, and generally have smooth surfaces. If the technology is mastered in the proper manner, these coatings can be produced with relatively low costs and high growth rates. These properties make DLC coatings suitable candidates to serve as a base layer for decorative coatings on relatively soft substrates. The problem related to adhesion between the DLC base coating and the decorative top coating has been mastered, as shown in a SVC presentation of 2008. The process parameters for depositing adequate DLC support layers on plastic substrates will be addressed in the current presentation.
Superhard amorphous hydrogenated carbon (a-C:H) coatings were prepared in a large scale batch coater equipped with 4 targets and electromagnetic coils. Coils around each target and an additional coil generating a magnetic field over the chamber volume caused high substrate ion currents. The sputter gas was a mixture of argon and acetylene. To improve the adhesion different interlayer systems, e.g. from chromium or tungsten carbide were used. The a-C:H coating deposition was carried out working with varying parameters of substrate bias (d.c. and pulsed d.c.), acetylene flows and coil currents. The highest hardness values were reproducibly measured with up to 50 GPa. The coatings characterization was done by SIMS and Raman spectroscopy as well as by hardness and wear tests. The hardness clearly depended on the hydrogen contents and the hardest coatings could be prepared at about 10 atom % hydrogen. Reasons for this behavior will be discussed considering the effects of the most essential process parameters. For a fully loaded machine processes suitable to deposit well adhering coatings were developed. To achieve optimum adhesion for components or tools, respectively, different types of interlayers were applied. The addition of HIPIMS steps allowed additional process modifications influencing coating properties, especially the adhesion.

Diamond-like carbon films (DLC) have proven to be excellent low friction and wear protection coatings for components and tools under different tribological conditions. The new generation of hydrogen-free ta-C films (ta-C = tetrahedral bounded amorphous carbon) have extraordinary high wear resistance under extreme abrasive conditions due to their hardness close to crystalline diamond. They possess excellent dry-running and emergency-operating properties at lubrication breakdown if their surface roughness adapt to the running conditions. Too rough ta-C films are very abrasive and the friction partner can be severely damaged in a short time. Conventional ta-C films deposited by unfiltered arc-deposition mostly are not directly applicable due to their typical high roughness. By using plasma-filtered deposition methods or polishing after deposition ta-C films with sufficiently smooth surface can be obtained. In this investigation, ta-C films are deposited with a pulsed arc deposition process (Laser-Arc) on different substrate materials. The ta-C surface roughness has been modified by three different methods. Firstly a mechanical polishing of the as deposited films, secondly the deposition of a soft running in top layer on the ta-C film and thirdly a new filtering process for ta-C film deposition were applied. The influence of these three methods has been studied by means of tribological testing under the same dry and lubricated friction condition. The results will be discussed and examples of successfully introduced applications of such modified ta-C films will be presented.
Wednesday, May 13

10:30 a.m. T-16 How Substrate Constraints Affect the Performance of Thin Film Coatings Under Dynamic Impact Loading

L.V. Davies, Caterpillar, Inc., Peoria, IL

Invited 40 min. Talk

The study of dynamic impact performance of thin film coatings has largely been focused on the coating alone, using high hardness, highly polished steel to eliminate substrate effects. But in practice, it is vital to study the combined effect of the coating and the substrate (known as the coating system). When selecting engineering steels for coated components a large amount of the decision is based on functionality, manufacturability and cost requirements, and less so on the tribological needs of the system. In sliding motion, this is less of a problem, provided the substrate has sufficient load bearing capability for the coating and operating conditions. However, under dynamic impact, substrate selection becomes much more critical. The cyclic impact loading applied to the coated surface generates energy waves that must be accommodated by the coating system. The substrate must possess a high toughness to absorb the severe macromechanical stress fields from the impacts whilst have sufficient elasticity to accommodate any substrate deformation that may occur under impact. This paper will define impact resistance and provide an example of how engineering functional requirements plus manufacture constraints directly affected the performance of a coated control valve.

Wednesday, May 13

11:10 a.m. T-17 Erosion, Corrosion and Wear Resistance and Microstructure of Diamond-Like Carbon (DLC) Coatings Prepared Using a Mesh Method

R. Wei, M. Jakab, V. Poenitzsch, and K. Coulter, Southwest Research Institute, San Antonio, TX

In this paper, we present a systematic tribological and microstructural study of diamond-like carbon (DLC) coatings deposited using a newly developed process. This process utilizes a metal mesh to enclose the parts. When a pulsed voltage of a few thousand volts is applied to the mesh, pulsed glow discharge is generated in the mesh to accomplish the deposition. This process does not only result in a high rate deposition of a DLC coating, but it also allows the deposition on non-conductive parts such as polymers and ceramics. Using this method, we deposited DLC and Si-DLC coatings on 1018 steel under various conditions. The microstructure of these coatings was studied using SEM and Raman sectroscopy. Their erosion resistance was evaluated using a micro sand blaster with 50µm Al₂O₃ particles at various back pressures at two incident angles, 30° and 90°. The coating corrosion resistance was evaluated using the polarization test, while the coefficient of friction and sliding wear resistance were studied using a pin-on-disc tribometer. For comparison purposes, a DLC sample was also prepared using the standard plasma immersion ion deposition (PIID) process. It was observed that the DLC deposition rate using the mesh method was over 10 times higher than that using the conventional PIID process. Most coatings showed higher resistance to erosion than uncoated 1018 steel. The coating also exhibited high polarization resistance, up to four orders of magnitude. In addition, all the DLC coated samples showed a coefficient of friction from 0.1-0.12 with negligible mass loss due to wear, in ambient conditions without lubricant. The research results show that this new method may be used for protection of low alloyed steel or low cost materials from wear, corrosion and erosion.
Although PVD is one of the most powerful technologies for coating, there are characteristics that PVD on its own can not impart in an efficient way because they intrinsically require a coating of a high thickness that can not be cost-effectively applied by PVD. Some of these properties are, for example, the adequate protection of the substrate from corrosion, or the capability to level the surface of the substrate to give a shiny bright appearance. Nowadays this functionality is being applied mostly with approximately 20 microns electroplated nickel. Nevertheless, due to the stresses involved in PVD, the parameters of this process must be carefully selected in order to obtain quality products in terms of adhesion. In this work, nickel plated substrates from four different suppliers have been coated with ZrCN by Cathodic Arc Evaporation. The study of the correlation between the stresses induced by the PVD process and the adherence achieved between the PVD layer and the nickel plated substrates has been carried out. For this purpose, coatings of different thicknesses were applied. Adherence was evaluated and residual stresses were calculated using the Stoney equation after measuring the curvature radius when applying the coating on very thin substrates. On top of that, a preliminary study of the nickel plated substrates has been carried out, characterizing the substrates by means of microstructure, through optical microscopy and SEM, roughness, through AFM, composition, by GDOES, microhardness and residual stresses.

Magnetron sputtered metal and ceramic films have poor adhesion to certain polymer structures especially polymethylmethacrylate (PMMA). Because of ion bombardment and short wavelength vacuum ultraviolet (VUV) radiation from the plasma, the PMMA surface suffers scission of the molecule’s side chain by degradation of the ester group, consequently weakening the mechanical properties of the polymer. Nanometer-scale metal oxide Al₂O₃ and TiO₂ films deposited by atomic layer deposition (ALD) were deposited on PMMA plates prior to magnetron sputtered Ti and TiC films. They proved to have a multifunctional role in enhancing the adhesion of the sputtered layer. The adhesion significantly improved with increasing ALD film thickness up to ~40 nm thickness at which point the adhesion strength reached the cohesive strength of the substrate material itself. Using infra-red spectroscopy (ATR-FTIR) to monitor changes in the bonding in the surface layers, it is shown that the ALD layers act both as a shield against ion bombardment and also against the effects of VUV radiation from the plasma and so prevents the structural changes in the PMMA which otherwise cause degradation of adhesion.
Optical Coating
Recent Advances in the Design of Optical Filters with Arbitrary Intermediate Refractive Indices

S. Larouche, Pratt School of Engineering, Duke University, Durham, NC

Invited 40 min. Talk

Most optical filters in use nowadays consist of a stack of layers of two materials with discrete refractive indices. At normal incidence, it has been demonstrated that such filters provide optimal solutions and it is possible to manufacture them with very good reproducibility. However, the use of graded-index layers or homogeneous layers with intermediate refractive indices might provide optimal solutions at oblique incidence and better mechanical properties. The recent years have seen rapid development in the processes used to fabricate thin films of mixed materials in controlled proportions with intermediate refractive indices. There is, therefore, interest for design methods exploiting those possibilities. This presentation reviews the advantages of using intermediate indices in the design of optical filters and the methods used to design such filters. It focuses on recent developments made at École Polytechnique de Montréal, namely the inclusion of two correction factors in the Fourier transform method to account for the effect of refractive index dispersion, and the step method, a new synthesis method specifically developed for the design of multilayer filters with intermediate indices. The challenges faced in the manufacturing of such filters are also discussed.

Antireflection Coating Design by Fourier Frequency Filtering with Frequency Range Adjustments

P. Verly, National Research Council of Canada, Ottawa, Canada

Frequency filtering is a useful Fourier optics technique also applicable to dielectric thin films. The Fourier relationship arises between the logarithm of a refractive index profile versus optical thickness and a function of its reflectance versus inverse wavelength (called the Q-function in the literature). This relationship allows decomposing the index profile in components corresponding to different regions of the spectrum. For example, one can construct complex reflecting filters from the superposition of simpler reflectors. Conversely it is possible to extract unwanted spatial frequencies from a known refractive index profile. An interesting application is the design of antireflection coatings by elimination of the Fourier frequencies from the AR band. An iterative approach based on this concept was proposed in the past. It was found that the Fourier calculations decrease the reflectance to a minimum after which it starts increasing again due to the inherent approximate nature of the Fourier approach in thin films. The present work shows that tuning the Fourier frequency range in the course of the design process results in significant improvements.
9:30 a.m. O-26 Synthesis Route to Garnet and Perovskite Thin Films via Quad-Reactive Co-Sputter Deposition of Amorphous Non-Equilibrium Alloy Oxides and Subsequent Annealing

D. Baldwin, 4Wave, Inc., Sterling, VA; M. Martyniuk and R.C. Woodward, The University of Western Australia, Perth, Australia; and R.D. Jeffery, Panorama Synergy Ltd., Balcatta, Australia

Amorphous layers of (BiDy)(FeAl)O$_{12}$ have been deposited at low substrate temperature by reactive plasma co-sputtering from four metallic targets and subsequently annealed to produce crystalline magneto-optical garnet films. Details of co-sputter deposition from bismuth, dysprosium, iron and aluminum metal targets by biased target sputter deposition (BTD) are presented. BTD, a form of PVD, uses a remote plasma source to provide sputtering ions and employs bi-polar pulsed DC bias with independently adjustable duty cycle for each target to control alloy oxide composition. Fully oxidized films of (BiDy)(FeAl)O$_{12}$ have been characterized for composition by Rutherford backscattering spectrometry (RBS) both before and after annealing. Film composition was easily controllable by BTD co-sputtering and remained constant after annealing. Post-annealing measurements by x-ray diffraction showed crystallinity with the main peak at ~32° indicating the garnet phase. SQUID measurements of magnetic anisotropy were made, optical absorption was characterized and preliminary results on Faraday rotation at 633 nm were obtained. A conclusion is that the demonstrated synthesis route for (BiDy)(FeAl)O$_{12}$ is robust, production-scalable and of general applicability, and the technique opens garnet, perovskite and other alloy-oxide systems to study in composition ranges not readily achievable by other means.

9:50 a.m. O-12 Multifunctional Optical Coatings on Polymers Deposited by Pulse Magnetron Sputtering and Magnetron Enhanced PECVD

P. Frach, H. Bartzsch, K. Taeschner, J. Liebig, and E. Schultheiss, Fraunhofer Institute for Electron Beam and Plasma Technology FEP, Dresden, Germany

Reactive pulse magnetron sputtering processes are of increasing interest for the deposition of high-precision optical coatings of high density, low roughness and shift-free environmental stability. SiO$_2$, Si$_x$N$_y$ and silicon oxynitride are attractive coating materials for interference filters produced by sputtering of a silicon target in a precisely controlled reactive gas mixture of oxygen and nitrogen. Polymer substrates are increasingly relevant for the application of optical coatings due to their mechanical and economical advantages. The opposing properties of hard and brittle metal oxide layers on one hand and of the soft and elastic polymer substrates on the other hand may lead to adhesion failure and crack formation of the coatings. Therefore it is necessary to adapt the coating properties to the surface characteristics of the polymer substrates by using adapted process parameters of the reactive pulse magnetron sputter deposition. Well-adhering multilayer or gradient layer stacks on polymer substrates are obtained even for thick coatings that fulfill not only optical requirements but have also diffusion barrier and hard coating properties. Magnetron enhanced PECVD using HMDSO and HMDSN as precursors allows to deposit carbon containing films with polymer-like properties. Results show the suitability of these coatings as hard coatings or matching layers.
Wednesday, May 13

10:30 a.m. O-13 On the Influence of Surface and Interface Roughness on the Optical Performance of Silver Based Stacks Deposited onto Plastic Film

R. Thielsch, R. Kleinhempel, T. Boehme, and A. Wahl, Southwall Europe GmbH, Grossroehrsdorf, Germany

Magnetron sputtered thin silver layers combined with various dielectric layers have found wide applications for solar control and heat protection in buildings and cars as well as in such challenging application like thin film EMI/NIR blocking filters in plasma display panels. The optical and electrical properties of various silver- metal oxide layer stacks were investigated in correlation with the deposition parameters. These properties are evaluated and correlated with the micro-structure of the coatings. Silver layers were combined with metal oxides like ITO, In2O3, Zn(Al)O and ZnSnO3 by magnetron sputtering (DC, unipolar pulsed) onto PET film. DMD stack sequence with up to 5 silver layers were deposited and analyzed by optical methods, thin film stress determination, measurement of the sheet resistance and by determination of structural properties (AFM, XRD). Measured optical data were compared with data from modeling of the stack optics which highlight the importance of the interface roughness on the performance. Another emerging application is for high reflectivity of concentrated solar reflectors on flexible organic substrates. The influence of interface roughness to the substrate on the overall performance is demonstrated.

Wednesday, May 13

10:50 a.m. O-14 Mo- and In-Doped VO2 Thermochromic Coatings Grown by Reactive DC Magnetron Sputtering

C. Batista, V. Teixeira, and R.M. Ribeiro, Department of Physics, University of Minho, Braga, Portugal

Sponsored Student Presentation

Thermochromic coatings are demonstrating a high potential to be applied as energy effective building and automotive window coatings due to their self-adaptative optical properties which actively modulate the solar radiation. VO2 is an example of a transparent thermochromic material which is a promising candidate for this application. The change on its optical/electrical properties takes place at 68°C as a result of structural transition. It has been demonstrated that substitutional doping of vanadium by other elements is effective in reducing the transition temperature, however, their effect on the structural and optical/thermochromic properties are not completely understood. In this study, we have prepared pure and doped VO2 thin films onto SiO2-coated glass substrates by reactive direct current magnetron sputtering. The chosen elements for the doping were molybdenum and indium. To our knowledge, it is the first time that indium has been used as a candidate to reduce the transition temperature of VO2 films. The films were characterized in terms of crystal structure and texture by x-ray diffraction (XRD) and the morphology of the surface has been analyzed and quantified by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The optical/thermochromic behavior of the films was studied by optical spectrophotometry in the UV-VIS-NIR range.
Wednesday, May 13

11:10 a.m. O-15 Optical Properties of ITO Thin Films Produced by Plasma Ion-Assisted Evaporation and Pulsed DC Sputtering

K. Fuechsel, U. Schulz, N. Kaiser, and A. Tuennermann, Fraunhofer Institute for Applied Optics and Precision Engineering IOF, Jena, Germany

Indium tin oxide is one of the most important transparent conductive oxides. Due to its high transparency in the visible spectral range and its metallic properties, ITO is used as transparent electrode, heat-reflecting mirror, or heatable layer. As a result of free and bounded electrons, a multi-oscillator model has to be applied to describe the optical properties from the UV to the NIR and to compute the extinction coefficient and the refractive index. This paper compares the results of reverse engineering for thin ITO films deposited by pulsed DC sputtering and by low temperature plasma ion-assisted evaporation. Design examples are shown to demonstrate the suitability of ITO as a high index material for optical coatings.

Wednesday, May 13

11:30 a.m. O-16 Low Temperature Deposition of Indium-Tin Oxide Films Using Magnetron Sputtering

J.I. Jeong and J.H Yang, Research Institute of Industrial Science and Technology, Pohang, Korea

By using a magnetron sputtering source, an indium-tin oxide (ITO) target was characterized in terms of sputtering conditions such as voltage-current relation, impedance change, change of deposition rate in DC and RF mode, respectively. In the DC mode, the deposition rate increased very rapidly when the power increased, whereas the change of the deposition rate was not so large when RF power was applied. The optimum process conditions have been employed to deposit indium-tin oxide (ITO) films on PET film substrate at low substrate temperature. Pretreatment by RF and pulse plasma and irradiation effects of halogen lamp on the properties of ITO films have been examined. Post annealing and UV radiation effects of the coated samples have also been investigated. Transmission electron microscopy (TEM) was employed to investigate the microstructure and crystallinity of the films. The transmittance and sheet resistance were investigated according to the deposition time. It has been found that the transmittance was highest and sheet resistance was lowest when the film thickness was in the range of 140nm. The pre-treatment of the substrate and post-treatment of the coated sample greatly affected the optical and electrical properties of ITO films as well as morphologies of the films. Systematic experimental results will be presented in this paper.
Cleantech Symposium
Wednesday, May 13

8:30 a.m. CT-5 Basic Economics of Photovoltaics for Vacuum Coaters

G. Smestad, Sol Ideas Technology Development, San Jose, CA

*Invited 40 min. Talk*

With widespread deployment of Photovoltaic (PV) power imminent, it is useful for researchers in the coatings sector to have at least a basic knowledge of the economic principles that govern PV modules and systems. Several simplified and illustrative equations are presented, along with a technical overview of the field. For the past several years, PV modules prices have deviated from 80% progress ratios exhibited since 1980. These ratios are obtained from experience or learning curves. Deviation is due, in part, to strong demand and a Si supply shortage that is expected to end within the next two years. Thin film PV modules are believed to allow for a rapid return to prior trends. If Balance of Systems (BOS) costs are considered for existing technologies, the near term (2015) cost of PV power could be 0.08-0.13 $/kWh, assuming a module of 15% efficiency that lasts at least 15 years under the irradiance levels found in the sunnier regions of the world. Although solar cells of 15% efficiency that last for 15 years can be competitive with fossil fuels, those of less than 8% efficiency with lifetimes < 15 years will likely not lead to competitive products at MW scales.

Wednesday, May 13

9:10 a.m. CT-6 A Turnkey Approach for CIGS Thin Film Photovoltaic Production

D. Schmid, I. Koetschau, A. Kampmann, and T. Hahn, centrotherm photovoltaics AG, Blaubeuren, Germany

*Invited 40 min. Talk*

centrotherm photovoltaics AG has chosen Cu(In,Ga)Se2 (CIGS) as material system for turnkey thin-film photovoltaic production lines because of its large potential concerning conversion efficiencies and economical production techniques. Several approaches are reported in literature for the deposition of CIGS thin-film solar cells, with two-stage processes being favoured by the industry. Here, a metallic Cu(In,Ga)-precursor is deposited, preferably by sputtering, while the crystallization of the CIGS-compound is completed by an annealing step under the presence of selenium. Within CIGS thin-film technology, centrotherm photovoltaics AG is introducing a completely new approach for the production of CIGS-layers, which has not been reported in literature so far. This centrotherm-unique new technology allows for both, an easy scaling of the preparation techniques involved as well as unrivaled short process intervalls for CIGS formation. Hence, the new technology developed by centrotherm photovoltaics AG is especially designed for the realization of economically operated in-line plants, since the fast processing of CIGS-modules is not restricted by module size. Thus, keeping the high efficiencies associated with the CIGS system, a considerable economical impact on thin-film solar cells due to this new technology is expected.
Wednesday, May 13

9:50 a.m. CT-7 TiO₂-Ge Nanocomposites for Solar Cells Applications

S.I. Shah and B. Ali, University of Delaware, Newark, DE; and M. Abbas, COMSATS Institute of Technology, Islamabad, Pakistan

Several new photovoltaic semiconductor materials and synthesis techniques have been developed as a result of the increasing need for renewable energy sources. Quantum dot (QD) based solar cell is potentially one of the best contenders. We have developed a thermodynamically stable nanocomposite (stable up to 900 °C) titania-germanium (TiO₂-Ge) which shows promise as the active layer for QD nanocomposite solar cells. In TiO₂-Ge nanocomposites Ge nanodots are distributed in a TiO₂ matrix. Due to the 3-D quantum confinement effect, tailoring of the optoelectronic properties is relatively easily done by simply varying the Ge nanodots size. Ge is particularly advantageous since the Bohr radius of Ge is relatively large, 25 nm. In addition to the specific results of the variation of the optoelectronic properties of TiO₂-Ge nanocomposites, some results on device fabrication and characterization will also be presented.

Wednesday, May 13

10:30 a.m. CT-26 Survey of Thin Film Solar Cell Technology in Germany

V. Sittinger, W. Diehl and B. Szyszka, Fraunhofer Institute for Surface Engineering and Thin Films IST, Braunschweig, Germany

Invited 40 min. Talk

Within the scope of limited non-renewable energy sources and the restricted capacity of the ecosystem for greenhouse gases and nuclear waste, sustainability is one important target in the future. Different energy scenarios showed the huge potential of photovoltaic to solve this energy problem. Nevertheless, in the last decade PV had an average growth rate of 44% per year. In 2007, the worldwide production of solar cells has grown to 4 GW. More than 90% of the recent production involves crystalline silicon technologies. These technologies have still a high cost reduction potential. On the other hand, the so-called second generation thin film solar cells based on a-Si/μc-Si, Cu (In,Ga)(Se,S)₂ or CdTe have material thicknesses of a few microns as a result of their direct band gap. Also the possibility of circuit integration offers an additional cost reduction potential. Nowadays, roughly 150 companies are engaged in this field worldwide. Especially in Germany, there are many companies focusing on thin film solar cells. Some of them have already started mass production. Schott Solar GmbH and Ersol Thin Film GmbH (takeover by Bosch GmbH) with the a-Si technology are collaborating in the development of a-Si/μ-Si tandem technology. The U. S. company, First Solar, produces in Germany using the CdTe technology. Würth Solar GmbH is also in production using the Cu(In,Ga)Se₂ technology. There are further different production lines under construction by companies for example by Avancis, Sulfurcell, Johanna Solar, and Solarion GmbH. Furthermore, the biggest company in the worldwide solar market so far is QCells Company, which has started up different subsidiary companies with all these technologies (Sontor, Calyxo, Solibro). Nevertheless, there are still many companies who have just started to build up their production lines. An overview of the research activity in different companies and with different thin film technologies will be given as well as different manufacturing and production processes.
A Novel Approach for Thin-Film Crystalline Silicon on Glass

K. Sharma, A. Illiberi, A. Branca, M. Creatore, and M.C.M. van de Sanden, Eindhoven University of Technology, Eindhoven, The Netherlands

Thin film technology is emerging as a new and promising approach for industrial production of PV modules: polycrystalline silicon (Poly-Si) films are fabricated by crystallization of amorphous silicon layers, grown on low-cost supporting materials (glass substrate). In this framework, we are developing a new approach for the production of Poly-Si thin films: the amorphous layers are deposited by using the expanding chemical vapor deposition (ETP-CVD) technique, which has previously demonstrated device grade a-Si:H at high deposition rate (7-11 nm/s). The crystallization process has been induced by means of Solid Phase Crystallization. Fourier Transform Infrared and Spectroscopic Ellipsometry (SE) diagnostics have been used to characterize both the as deposited and annealed a-Si:H layers, by measuring the thickness, hydrogen content, bonding configuration and optical constants of the films. The imaginary part of the pseudo-dielectric function has been measured by means of SE to give insight into the crystallization degree of the annealed a-Si:H films. The results have been confirmed by Raman diagnostics. The structural material quality of the Poly-Si films has been investigated by means of cross-section Transmission Electron Microscopy: crystal grains (1 um lateral dimension) extending over the entire thickness (1um) of the annealed a-Si:H films on glass have been observed.

Progressive DC Power for TCO Deposition

D. Ochs, HUETTINGER Elektronik GmbH + Co. KG, Freiburg, Germany; and P. Rozanski and P. Ozimek, HUETTINGER Electronic Sp. z O.O., Zielonka, Poland

Transparent conductive oxides (TCO) as ZnO:Al (AZO) are of great importance as transparent electrical contact layer for thin film solar cell application. As this material has a very high arcing rate pulsed DC power processes have been needed in the past. A new DC power supply family has been developed with the goal to replace these expensive pulsed DC processes by standard DC processes. The most important feature of this power supply is a very fast and advanced arc management. After detecting an arc a positive voltage is applied to the cable between power supply and cathode. This compensates the stored energy of the cable and reduces the energy supplied into the arc after power switch off. Measurements using a 60kW DC power supply have shown very stable process conditions over a long time for planar and tube AZO targets. Residual arc energies significantly less than 1 mJ/kW could be calculated from oscilloscope measurements of the voltage and current behavior during the arc event. Because of the very short process interruption stable processes with the in production typical arcing rates of several thousand arcs per second become possible. Transmission and resistivity of layers deposited with DC have been measured and compared with pulsed DC deposited layers. Pure DC deposited layers show similar or even better results in respect of transmission and resistivity compared to the pulsed deposited layers. It could be demonstrated that pulsed power can be replaced without any disadvantage.
In flexible organic electronic devices, the high brittleness and limited mechanical ruggedness of requisite inorganic thin films are a likely source of failure. It is, therefore, important to understand and improve the mechanical limits of these functional layers. In the present study, a multiple film cracking in thin film/substrate composite systems was analysed. Specifically, the experimental measurement of multiple cracking of indium tin oxide films (ITO) deposited by reactive magnetron sputtering on polyethylene terephthalate (PET) substrates was investigated. In addition, thin homogeneous and highly transparent films of the conductive polymer poly(3,4-ethylene dioxythiophene) (PEDOT) were prepared by means of spin coating via moderator controlled in situ chemical oxidative polymerization of EDOT on the ITO coated PET sheets. Such multilayered films may be used as cathodically colouring electrochemical half cells for the assembly of flexible electrochromic devices. The system was subjected to an unidirectional tensile loading. A shear lag model was used to derive the stress distribution in the system, and the film cracking problem was analysed using the strength criteria. In addition, a numerical computer simulation was performed to simulate the number of cracks, the crack distribution and the film fracture strength using a Weibull statistical analysis. The simulation predicted successfully the crack density and the distribution of fragment lengths during the progress of multiple cracking. The influence of the polymeric top layer on the mechanical properties of the composite systems is discussed.
Wednesday Afternoon
May 13, 2009
Wear protecting PVD coatings were first introduced on a large scale to automotive applications in the early eighties; AlSi20Co nanocomposite coatings on conrod bearings were the materials enabling the introduction of the first turbocharged diesel engines. But the big wave started with the introduction of the WC/C coatings for the new hydraulic cam followers of the common rail system by Balzers/Bosch/INA. From there on, the application of PVD coatings spread quickly to a wide range of components. PVD coatings for automotive components have become the fastest growing sector of PVD wear protection coatings and the main enabler of innovations in cars. Today PVD coatings can be found in almost every system of a car. This tutorial will present the requirements and wear mechanisms of the most important subsystems of cars and compare them with the PVD coatings currently used. A special focus will be given to the recent and current developments for engine components.
Process Modeling and Control
Wednesday, May 13

1:30 p.m. A-1 Fundamental Understanding of Pulsed PECVD Through Diagnostics and Modeling

C.A. Wolden, Colorado School of Mines, Golden, CO

Invited 40 min. Talk

Atomic layer deposition (ALD) is the leading technology for nanoscale synthesis of thin films, imparting digital control over thickness and composition, uniformity, and conformality. However, its low rates preclude its use for the practical synthesis of relatively thick mesoscale coatings (100 - 1000 nm). There are tremendous opportunities in this area, and some applications of interest include flexible electronics, precision optical components, thin film batteries, and solar cells. At CSM we have developed low frequency (~1 Hz) pulsed plasma-enhanced chemical vapor deposition (PECVD) as an alternative to ALD for self-limiting growth of metal oxides. Pulsed PECVD retains Angstrom level control over thickness and composition, but at significant net rates (> 30 nm/min). In this presentation will introduce the principles of the technique, highlighting a number of salient results. Pulsed PECVD appears to have widespread applicability, having been successfully used to synthesize a wide variety of oxides (ZnO, TiO2, Al2O3, Ta2O5). Achievements include demonstration of high rates, exceptional quality, a high degree of conformality, and room temperature synthesis of crystalline semiconductors. The technology continues to expand and current efforts in the synthesis of nanolaminates, mixed metal oxides, and inorganic/organic hybrids will be described. To date, pulsed PECVD process development has been largely empirical. In order to continue to expand and refine this deposition technology for emerging markets it is clear that a more fundamental understanding of the process is necessary. We have embarked on a project that employs time-resolved diagnostics (Langmuir probe, optically emission spectroscopy (OES), electrical impedance spectroscopy (EIS)) to provide fundamental insights into the dynamics controlling the plasma chemistry in these systems. These in situ measurements are being used to validate state-of-the-art computational models of the process. In pulsed PECVD the role of both neutrals (fluid flow, transport phenomena, gas- and surface chemistry) and activated species (ion, electrons, metastables) are of comparable importance. As such, this system poses unique challenges to modeling that will be addressed through transformative approaches to accelerate computations for plasma simulations with large number of species and chemical reactions. In this talk we will describe the development of this integrated tool set, as we work to create a platform that will enable automated process development and provide strategies for real time control.

Wednesday, May 13

2:10 p.m. A-2 Optimizing Sputter Processes via 3D Magnetic and Plasma Simulations

D. Monaghan, M. Holik, and V. Bellido-Gonzales, Gencoa Ltd., Liverpool, United Kingdom; and J. Bradley, Liverpool University, Liverpool, United Kingdom

The presentation will demonstrate how the performance of a magnetron sputter cathode in terms of target use, uniformity and coating quality, can be greatly enhanced by appropriate simulation tools. All geometries of magnetron sputter targets rely upon the nature of the magnetic field design to optimize the plasma distribution and hence the target erosion. Simple circular geometries can be solved by 2D simulations whereas rectangular geometries require 3D solutions. Magnetic field solutions in 3D are readily available from a range of commercial packages. A 3D magnetic field simulation goes some way to providing the ability to create a good magnetic trap. To provide a replication of the resulting target erosion requires the collisional and anodic effects to be taken into account. This requires a 3D plasma simulation and calculation of the impact points of the particles on the target surface. It will be shown that 3D magnetic and plasma simulations can provide an accurate prediction of how a target erodes. This in turn leads to the ability to improve the sputter process without exhaustive plasma test and results in higher efficiencies. The plasma modeling also predicts the magnetic changes required to tune uniformity and the coating properties and a number of examples will be shown.
2:30 p.m. A-3 Simulation Analysis of Plasma Discharge Anomalies in PVD and PECVD Processes

M. Siemers, A. Pflug, and B. Szyszka, Fraunhofer Institute for Surface Engineering and Thin Films IST, Braunschweig, Germany

Developing industrial PVD and PECVD plasma sources within the framework of a model based simulation environment has been subject of large R&D efforts in the past decade. However, substantial progress has been possible only within the last few years due to the increasing availability and utilization of parallel hard- and software architectures. With the use of massive parallelization we realized a full featured 3D simulation environment for low temperature plasma discharges based on the Particle-in-Cell Monte Carlo (PIC-MC) approach. A finite element mesh model has been incorporated to enhance geometry mapping into the simulation domain. Within the simulation environment all relevant static and dynamic process parameters of a plasma discharge (e.g. pressure distribution, magnetic field and coater geometry) can be varied. This avoids costly touch ups and aberrations during the design phase of a coater and gives insights into the complexity of 3D plasma discharges which are hard to achieve using conventional experimental techniques. Also, the plasma simulation turns out to be a rather cost effective design tool since costly expenses for plasma diagnostics can be avoided using the process model. We are going to show simulation results regarding discharge anomalies in PVD and PECVD processes. The visualization of macroscopic and numerical process quantities helps us identifying the cause of unwanted plasma aggregations, e.g. from insufficient magnetron and compartment layout respectively.

2:50 p.m. A-9 Advanced Radio Frequency Plasma Enhanced Evaporation and its Advantages

W. Schwärzler, Provac AG, Balzers Principality of Liechtenstein and D. Gary, Ricmar Sales and Service Inc., Aptos, CA

Dielectric layers functionality, complexity, and stability demands are ever increasing. Efficient coating layers easily can be produced by using Advanced Radio Frequency Plasma Enhanced Evaporation techniques in high vacuum environments. This presentation gives a brief overview about enhancing conventional E-Beam evaporation with the Advanced Radio Frequency Plasma Techniques evolving from Taurion technology. Advanced Radio Frequency Plasma Enhanced-Electron Beam Evaporation (RFPE-EBE) is predominately used to produce absorption free and densified oxide layers such as SiO₂, TiO₂, Ta₂O₅, Nb₂O₅, and nitrate metallic layers (possible to produce hard layers-nitride basis). The bombardment of the substrate with high-energy ions of the respective process gas can also be used for degassing of the substrate surface and increasing the cleaning effect. Advanced Radio Frequency Plasma Technology can directly be used in combination with existing coating processes. Advantages of RFPE-EBE include easier operations, maintenance, cleaning, and use of the basically neutral plasma of the high-frequency plasma sources that renders damage to the substrates by electrical discharges impossible right from the start. Additionally, the use of pure process gases expands new areas of applications, where the other processes are not suitable.
Wednesday, May 13

3:30 p.m. A-7 The Modeling of Large and Distributed Vacuum Systems

J. Luby, Edwards Vacuum, Tewksbury, MA; A. Chew, Edwards Vacuum, Crawley, United Kingdom; and M. Galtry, Edwards Vacuum, Shoreham, United Kingdom

As the size and complexity of vacuum systems increases, the financial and technical challenges do so also. Accurate modeling allows system optimisation in advance of engineering efforts and negates the need for inefficient empirical iterations. This paper will describe a computational modeling technique which allows the modeling of vacuum systems incorporating any number of primary and secondary pumps and all other elements of a vacuum system. Real examples will be used to illustrate accuracy and efficacy.

Wednesday, May 13

3:50 p.m. A-6 Differential Sputter Yield Measurements of Single- and Multi-Element Targets Due to Ion Beam Bombardment

A. Yalin, B. Rubin, J. Topper, and C. Farnell, Colorado State University, Fort Collins, CO

Detailed knowledge of differential sputter yield profiles could aid the optimization of coating processes based on ion beam etching. In this contribution we present an experimental setup and demonstrative results for measurement of differential sputter yield profiles (i.e. angular profiles of ejected particles). The facility employs a gridded ion source to provide an approximately mono-energetic and collimated beam. Sputter yields are measured using a quartz crystal microbalance (QCM) deposition monitor. We present measurements of differential sputter yield profiles for molybdenum targets, and boron nitride targets, due to bombardment by xenon ions in the energy range of 100-300 eV. Total sputter yields (found by integrating the differential profiles) are validated against weight-loss measurements and published values. The measured profiles are fit with Modified Zhang expressions to compactly describe the results using two fit parameters, a characteristic energy (E*) and total sputter yield (Y). We also present demonstrative modeling that uses the ion beam conditions and differential sputter yield profiles to model and optimize placement of targets and substrates in coating processes.
In many industrial applications, strain gauges are commonly used for measurements of applied forces or the loading status of work pieces. While commercial strain gauges using polyimide foils can cause errors due to influence of humidity, thin film strain gauges are of interest for direct application on the work piece surface. Besides the improvement of the signal due to avoiding glue and polymer substrate, the gauge factor can be further improved by using new sensor materials. For diamond-like carbon films, a high gauge factor up to 1000 is published in literature. Unfortunately, a very high negative temperature coefficient of resistance TCR is also connected with those properties. By adding metal with typically positive TCR, thermal compensation in DLC can be realized. An investigation was conducted of suitable material for thermal compensated strain gauges with a gauge factor higher than two, as is typical for conventional NiCr. DLC films were doped with different metals (Ti, W, Ni) and the resulting sensing properties were measured. For Ni-DLC, the TCR showed a zero crossing. The gauge factor of those films was higher than ten, resulting in a significant increase in sensitivity with the potential for thermal compensation.
Vacuum Web Coating
Wednesday, May 13

1:30 p.m. W-8 Roll-to-Roll Deposition for OLED Lighting Devices

C. May, S. Mogck, and J. Amelung, Fraunhofer IPMS, Dresden, Germany

OLED technology has successfully penetrated the display market within the last years. To enable OLED technology for further markets like signage and general lighting, cost reduction is a major issue. The use of ITO coated glass substrates as used for passive matrix displays is therefore not an option for lighting application. Low cost metal foil as substrate for the deposition of organic light-emitting diodes will allow a major cost reducing step. Furthermore, there is a need to replace sheet to sheet fabrication technologies by roll-to-roll-processing. Using the existing equipment for vacuum evaporation of small molecule materials on glass substrates OLED on commercial aluminum sheets were fabricated. Relative thick doped transport layers allow it to get stable devices on rough substrates too. Results for highly efficient top emitting OLEDs on rough metal substrates will be presented. Within the Center of Organic Materials and Electronic Devices Dresden, (CÖMEDD) of Fraunhofer IPMS a roll-to-roll line for research and development in OLED lighting is currently under installation. The paper discusses the line concept and technological demands for OLED roll-to-roll fabrication as substrate roughness, substrate patterning, small molecule organic layer deposition and encapsulation. First results will be presented.

Wednesday, May 13

2:10 p.m. W-9 Opportunities and Challenges in Flexible Electronics

D.J. McClure, 3M Corporate Research, St. Paul, MN

Invited 40 min. Talk

The promise and/or attractiveness of flexible electronics seem obvious: building electronic devices in roll-to-roll formats would reduce device costs dramatically. Some applications actually require flexibility. There are likely more applications that do not require flexibility per se, but rather value the reduced weight, reduced thickness, and, perhaps most importantly, the improved mechanical robustness that flexibility provides. We will review the many technical challenges involved in flexible electronics: materials performance, durability, and cost, substrate cleanliness and distortion issues, device patterning and yield problems, and the lack of standardized production tool sets. Many opportunities for materials and process advancements remain. A key barrier to rapid expansion of this technology area is the lack of an identifiable market that justifies the capital investment required without incurring a disproportionate market risk.
2:50 p.m. W-10 Pentacene Deposition for Vacuum Web Coated Organic Transistors

G.A.W. Abbas and H.E. Assender, University of Oxford, Oxford, United Kingdom

We have investigated routes to the development of roll-to-roll vacuum processed organic TFTs. Use of a vacuum-deposited organic layer as the semiconductor in a transistor device could allow the integration of this deposition with other vacuum-deposited layers with a view to cheap, flexible TFT arrays. One candidate material for the semiconductor is evaporated pentacene, and much preliminary work has been reported by many groups in constructing devices on rigid substrates under high vacuum static deposition conditions. In an attempt to investigate a roll-to-roll deposition (with a lower vacuum than previous studies) and to implement a vacuum compatible surface functionlisation method, we investigated the structure of pentacene films thermally grown at low vacuum with various gas ambience conditions (3x10^{-3} mbar of N₂ and Ar). The semiconductor films exhibited a thickness-driven structural transformation which controls the conductivity of the layer. X-ray diffraction and micro-focal Raman spectroscopy results will be presented to show how the vacuum conditions control the quality of the deposited semiconductor layer. These results imply that the optimal charge transport efficiency in pentacene semiconductors can be obtained through the appropriate growth environment.

3:30 p.m. W-11 Sputtering Synthesis of Antimicrobial Materials and Associated Process Optimization Approaches to Remain Competitive in Global Markets

D. Field, NUCRYST Pharmaceuticals, Fort Saskatchewan, Canada

Invited 40 min. Talk

While silver has long been known to possess antimicrobial properties, the most commonly used forms (silver nitrate and silver sulfadiazine) have historically demonstrated serious shortcomings in terms of efficacy and delivery. In the 1990s, magnetron sputtering was used to synthesize unique nanocrystalline antimicrobial silver films, which addressed these shortcomings. The improved performance of these films resulted in rapid market penetration of various sputter-coated, silver-based products for treatment of serious burn wounds and other indications. Following on this success, sputtered silver-based powders were also developed for use as antimicrobials and anti-inflammatories. This presentation will describe the development and efficacious properties of these sputtered, silver-based antimicrobial films and will discuss the challenges in synthesizing such materials on a wide variety of substrate types. Attention will be given to the various approaches in dealing with such challenges so as to remain competitive in a global market. More specifically, the reduction of manufacturing costs through various theoretical and experimental approaches will be reviewed. Examples of specific challenges, such as limiting heat flux at the substrate, will also be presented.
4:10 p.m. W-12 In-Register In-Vacuum Pattern Printing; From Wish to Reality

N. Copeland and L. Harland, General Vacuum Equipment, Heywood, United Kingdom (Presented by A. Jack, General Vacuum Equipment, Heywood, United Kingdom)

The use of flexo printed oil within a metallizer is a well known method that enables complex patterns to be metallized directly in a single pass. In this new development this in-vacuum pattern printing process has been developed further, so that the positioning of the pattern can be controlled to remain in-register with precision to a printed or embossed mark on the polymer substrate. This means that a pre-printed or embossed substrate can now have selective metallization applied to compliment existing designs. This is an important development as it assists the production of advanced packaging designs such as high security devices that depend on both the clear and un-metallized areas be in-register to highlight the different security features. In this paper we will highlight the benefits of this technology including examples of material that has been metallized in-register.

4:30 p.m. W-13 Impact of Metallizing Process Parameters on Metal Deposition, Optimum Film Properties and Converting Performance

E. Mount, EMMOUNT Technologies, LLC, Canandaigua, NY

The metallization process is straightforward and conceptually well understood, but at the same time it is quite complex and at times troublesome. While the overall process parameters for each machine are essentially the same, we find that various chambers will show an increased sensitivity to some parameters as opposed to another set of parameters. The controlling factors for each machine will impact the quality, metallized properties and the converting performance of the final product. As evidence of this, we find metallization chambers which can readily produce a product that the metallizer down the street just cannot reproduce and vice versa. The focus of this talk will be to review the principle metallizing process parameters which are common for all metallizers, to compare those that are specific to unsupported and supported machines and explore how these process parameters can impact the metallized film properties and defects. In some cases, comments relating to the film design and its impact on the metallizer process and defect generation will also be made.
Aluminum foil has long been the dominant material used for applications requiring low emissivity surfaces for heat reflection and energy management. Metallized aluminum films, though initially exhibiting emissivity values equal to their bulk foil counterparts, are far more susceptible to performance degradation as a result of oxidation and corrosion of the reflective aluminum layer in the presence of heat and moisture. This is due in part, but primarily, to the amount of sacrificial aluminum available to be consumed by the oxidation reaction. Investigated are novel, multi-layer, low emissivity materials incorporating one or more vacuum deposited layers designed to protect vacuum deposited aluminum layers from oxidation and corrosion, as well as to enhance the emissive properties of the multi-layer structure by providing a planarized surface on which to deposit the reflective aluminum layer. Various substrates ranging in material composition and breathability are investigated as potential replacements for perforated, and non-perforated aluminum foil laminates for heat reflection and low emissivity applications.
High Power Impulse Magnetron Sputtering
HIPIMS
**Wednesday, May 13**

**1:30 p.m. HP-8 Analysis of Reactive HIPIMS Discharge Based on Time- and Space-Resolved Optical Emission Spectroscopy**

M. Hala, N. Viau, O. Zabeida, J. Klemberg-Sapieha, and L. Martinu, École Polytechnique de Montréal, Montréal, Canada

*Sponsored Student Presentation*

HIPIMS has recently been applied to the fabrication of metal nitrides and oxides. However, detailed gas phase processes in reactive HIPIMS discharges are not yet well understood. In the present work, we employ high resolution Optical Emission Spectroscopy (OES) to perform a systematic time- and space-resolved study of the plasma in pure Ar and different N\textsubscript{2}(10-90%)/Ar mixtures excited by high-power pulses at a frequency of 50 Hz and a duty cycle of 1%, applied to a chromium target. The monitoring of both neutrals (Ar\textsuperscript{0}, Cr\textsuperscript{0}) and ionized species (Cr\textsuperscript{+}, Cr\textsuperscript{2+}, N\textsubscript{2}\textsuperscript{+}) during each pulse revealed two distinct discharge phases (like in the case of sputtering in pure Ar), namely: Phase 1, dominated by signals from Ar and N\textsubscript{2}, followed by Phase 2 dominated by emission from Cr neutrals and ions. The presence of Phase 2, earlier attributed to self-sputtering, is clearly distinguishable by the characteristic shape of the target current waveform for any N\textsubscript{2}/Ar ratio. Its duration depends on the applied cathode voltage and the working pressure. The addition of N\textsubscript{2} into the discharge at the same cathode voltage and at the same total pressure leads to a 2.5-fold increase in the peak current and up to a tenfold increase in the metal ion-to-neutral signal ratio. The observed phenomena are correlated with the coating's microstructure and composition.

**Wednesday, May 13**

**1:50 p.m. HP-9 A Langmuir Probe Study of the Plasma Parameters in the HiPIMS Discharge**

J.T. Gudmundsson, University of Iceland, Reykjavik, Iceland; and P. Larsson, D. Lundin, and U. Helmersson, Linköping University, Linköping, Sweden

We describe measurements of the plasma parameters in a high power impulse magnetron sputtering (HiPIMS) discharge. A Langmuir probe is used to determine the plasma parameters, such as the electron temperature, the electron density, the floating potential and the plasma potential, as well as the electron energy distribution function (EEDF). The spatial and temporal variation of the plasma parameters and electron energy distribution function are recorded in the pressure range 3 - 20 mTorr. The electron density peaks at \(5 \times 10^{18} \text{ m}^{-3}\) for 40 - 80 mm distance from the target surface for all pressures investigated. The electron temperature reaches its peak value of 1.5 - 3 V at 80 \textmu s after pulse initiation, in the pressure range 5 - 20 mTorr. The plasma potential and the floating potential peak towards the end of the pulse. We will in particular explore the EEDF and the spatial variation of the plasma potential at various times during the discharge.
High power impulse magnetron sputtering (HIPIMS) is a novel technique successfully implemented on full scale industrial machines. HIPIMS utilizes short pulses of high power delivered to the target in order to generate high amount of metal ions. The life-span of ions between the pulses and their energy distribution could strongly influence the properties and characteristics of the deposited coating. In many modern industrial coating machines the sample rotates on the sample holder and changes its position and distance with regard to the magnetron. Time resolved measurements of the ion energy distribution function (IEDF) at different distances from the magnetron have been performed to investigate the life-span of ions at various distances from target. The measurements were performed using two pressures, 0.9 Pa and 3 Pa to investigate the influence of working gas pressure on IEDF. Plasma sampling energy-resolved mass spectroscopy was used to measure the IEDF of Ti$^{1+}$, Ti$^{2+}$, Ar$^{1+}$ and Ar$^{2+}$ ions in HIPIMS plasma discharge with titanium (Ti) target in Ar atmosphere. The measurements were done over a full pulse period and the distance between the magnetron and the orifice of the mass spectrometer was changed from 25 mm to 215 mm. At shorter distance between target and the mass spectrometer we measured higher energies and shorter life-spans of the time averaged IEDF measurements, e.g. as distance increased from 100 mm to 210 mm, the life-span increased from 5 ms to 10 ms. This effect is emphasized at higher pressure. Low pressure measurements exhibit a higher energy tail compared to high pressure both for metal and gas IEDFs, at 150 mm distance high energy tail of Ti$^{1+}$ spans up to 100 eV at low pressure compared to only 30 eV at high pressure.
Wednesday, May 13

2:50 p.m. HP-12 Arbitrary Voltage Pulse Shape Plasma Generator with RF Capabilities for Material Processing

R. Chistyakov and B. Abraham, Zond Inc./Zpulser LLC, Mansfield, MA

A new arbitrary voltage pulse shape plasma generator with capabilities to generate RF discharge was developed for RF superimposed high power pulse magnetron sputtering. Plasma generator consists of two units; arbitrary voltage pulse shape unit and an RF unit. A special designed RF filter prevents RF power to penetrate inside the arbitrary voltage pulse shape unit. An arbitrary voltage pulse shape plasma generator with RF discharge capabilities gives unique opportunity for controlling plasma parameters. The principals of operation of new arbitrary voltage pulse shape plasma generator with RF generation capabilities will be presented. Method of generating multi step voltage pulses in the presence of RF discharge will be discussed.

Wednesday, May 13

3:30 p.m. HP-13 Reactive Sputtering Using HiPIMS

M. Aiempanakit and E. Wallin, Linkoping University, Linkoping, Sweden; W. Moller, Forschungszentrum Dresden-Rossendorf, Dresden, Germany; and U. Helmersson, Linkoping University, Linkoping, Sweden

Reactive sputter deposition of various oxides using HiPIMS have shown that the process becomes surprisingly stable and that hysteresis effects can be reduced and in some cases completely avoided. We have shown this for the case of deposition of Al$_2$O$_3$. Modeling of the reactive process, using a modified version of the Berg model, shows that this hysteresis-free behavior can be reproduced by setting the target etching in the model to a level that corresponds to what is expected during the HiPIMS-pulse. This indicates that the oxide build-up on the target in-between pulses is very small. A significant reduction of target poisoning is also confirmed by in situ target analysis. Here we present results also from deposition of CeO$_2$ which show a similar result. However, shorter pulse-off times are required for elimination of the hysteresis effects, indicating a slightly more rapid oxide build-up in-between the pulses for this material. Based on these results, the origin of the process stabilization as well as routes for optimization of the process conditions will be discussed.
3:50 p.m. HP-14 Progress in Process Control of High Rate HiPIMS Processes for Optical Thin Films

M. Vergoehl, O. Werner, and S. Bruns, Fraunhofer Institute for Surface Engineering and Thin Films IST, Braunschweig, Germany

It is well known that HPPMS/HiPIMS processes yield lower deposition rates due to the backscattering effect of positively charged metals ions to the target. In addition, for the deposition of isolating materials as Al$_2$O$_3$ or SiO$_2$, standard HiPIMS processes suffer from low process stability and arcing due to the low frequencies. Recently we developed a process where with bipolar, unipolar mid-frequency pulses is superimposed to the HiPIMS pulses. This yields a higher stability in the case of highly isolating oxides compared to the superposition of a DC signal. In addition, for pure HiPIMS, the control of the oxygen partial pressure turned out to be important to obtain high quality ZrO$_2$ films. In the present paper, a superimposed bipolar MF-HiPIMS, which in addition can be controlled at arbitrary set points of the oxygen partial pressure, was realized. With this process, highly isolating films like of Al$_2$O$_3$ and SiO$_2$ can be deposited from metallic targets at high rates and on larger areas. Optical and other properties of the films as stress, hardness and surface roughness deposited with the mentioned processes will be presented and discussed.

4:10 p.m. HP-15 Heat Treatable TCO Film for Position 1 Based on HIPIMS

V. Sittinger, F. Horstmann, W. Boentoro, W. Werner, and B. Szyszka, Fraunhofer Institute for Surface Engineering and Thin Films IST, Braunschweig, Germany

Functional coatings on weather exposed surfaces produce a huge interest in the architectural glazing industry. Since these coatings are exposed to atmospheric conditions and natural environment e.g. dust, dirt or salt, their resistance to mechanical abrasion and their chemical stability has to be ensured to guarantee their function over lifetime. The need of a cost-effective process for curved and toughened glass necessitates the use of float glass and a heat treatable and bendable coating. Well insulated glass units (Ug < 1.0 W/m$^2$K) mist up at a clear night to a greater or lesser extent depending on the glazing concept and its mounting angle. Mist and water condensation on glass surfaces can disturb the view. A Low-E coating at the outer glass surface such as transparent conductive oxides (TCO) can almost entirely prevent water condensation on its surface. A TCO coating has been developed for position 1 based on a high power impulse magnetron sputtering (HIPIMS) generator. A prototype power supply has been used for depositing ITO films at varying deposition parameters. Structural analysis has been carried out for films with different deposition parameters before and after heat treatment. Film properties have been studied by optical spectroscopy (UV/VIS), atomic force microscopy (AFM) and x-ray diffraction (XRD) measurements. Mechanical stability has been tested with taber-, scratch-, and sand trickling tests. The impacts have been observed with light and scanning electron microscopy as well as haze measurements. Furthermore, the results are compared with conventional DC sputtered ITO films and evaluated in respect to lifetime stability as well as coat and bend applicability.
Modulated pulse power (MPP) sputtering is a variation of high power pulse magnetron sputtering that overcomes the rate loss issue through modulation of the pulse shape, intensity, and duration. Usually MPP uses a two-step voltage pulse to create highly ionized magnetron plasma. The first voltage step creates a low power magnetron discharge, and once it is stable the cathode voltage is modulated with an increase of this voltage, resulting in the generation of high power magnetron discharge and strongly ionized plasma. The pulse shape and duration and plasma perturbations directly affect the degree of ionization of the sputtered material. In this study, silicon, silver and reactive ITO films were deposited with the modulated pulse power sputtering approach. The applied voltage pulse shape to the magnetron generated a high power pulse discharge and directly affected the degree of ionization of the sputtered material. Nanometer scale layers of silicon, silver and reactive ITO films were alternately deposited, and the thickness and structure of each nanolayer was controlled by varying the output voltage pulse shape of the MPP plasma generator. The OES of plasmas, film structure, orientation, and surface roughness were analyzed and measured. The data will be presented.

High Power Impulse Magnetron Sputtering (HIPIMS) is an exciting technique to produce a highly ionized sputter flux for film densification, surface modification, trench filling, adhesion promotion and other applications. Layers produced with HIPIMS show superior properties in many applications. The most investigated and promising HIPIMS application is for hard coatings in wear and corrosion protection. HIPIMS significantly changes the hysteresis curve in reactive sputtering, offering much higher deposition rates of compound thin films. Also the HIPIMS process has a significantly lower substrate heat load than standard magnetron sputtering enabling high rate coating even on temperature sensitive substrates. HIPIMS power supplies can be added to existing sputter systems with little or no system modification making them attractive as a way of extending process capability. However the HIPIMS parameters of pulse width, pulse frequency, pulse current, and pulse voltage are completely different from what needs to be specified with more conventional types of supply. This means that when first choosing a HIPIMS power supply it can be difficult to know what to specify. This paper describes the process effects of these parameters, and looks at their typical values. A simple and general step by step guide is given to specifying a HIPIMS power supply for sputter systems.
Cleantech Symposium
Wednesday, May 13

2:30 p.m. CT-11 Emissivity Modulating Electrochromic Device

H. Demiryont, Eclipse Energy Systems, Inc., St. Petersburg, FL

*Invited 40 min. Talk*

Eclipse infrared electrochromic device, IR-ECD, is an all-solid-state, all inorganic vacuum deposited monolithic thin film system functioning as an electrically controlled dimmable mirror in IR region. Maximum reflectance corresponding to bleach condition of the system is around 90% (low-e condition, e=0.1). The minimum reflectance reaches near zero in the colored condition of the system (high-e, e=1). Average emissivity modulation of Eclipse IR-ECD is 0.8 in the 8-12 micron region. Emissivity modulation is associated with reversible tuning properties of the system at 3 to 5 and 6 to 9 micron regions. Tuning properties of the system are associated with the cooling/heating of the IR-ECD surface. Because of the light weight (5g/m²), low voltage requirement (±1 Volts), extremely good emissivity control properties around room temperature region and controllable process ability on any substrate by reliable thin film deposition process makes this technology very attractive for satellite-spacecraft thermal control applications. Functionality of Eclipse IR-ECD has been successfully tested in space since March 8, 2007. This paper presents recent developments on the IR-ECD including space functionality test results. Space environmental tests will be reviewed in the paper.

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Wednesday, May 13

3:30 p.m. CT-12 New Thermochromic Coatings for Energy Efficient Windows

C.G. Granqvist, N.R. Mlyuka, and G.A. Niklasson, Ångström Laboratory, Uppsala University, Uppsala, Sweden

Thermochromic materials change their transmittance reversibly at a "critical" temperature. Vanadium dioxide films are well known and have a larger infrared transmittance below the critical temperature than above it. Applications on energy efficient windows have been hampered by two things: the critical temperature is too high (about 60°C), and the visible transparency is too low for thicknesses giving good thermochromism. These problems have remained for decades despite much research. This paper shows that magnesium doping increases the visible transmittance substantially and also decreases the critical temperature to a comfortable temperature. Doping with aluminium gives enhanced transmittance but increases the critical temperature somewhat. Specifically, we report recent data on vanadium dioxide films doped with Mg, Al, W, and combinations of these. We believe that these results make thermochromics more viable for window applications. Furthermore, new combinations of electrochromic and thermochromic coatings lead to "smart" windows with new and superior functionality.
Wednesday, May 13

3:50 p.m. CT-13 Boron-Doped Polycrystalline Diamond Electrodes for Electrochemical Applications

M. Becker and T. Schuelke, Fraunhofer USA, East Lansing, MI; G. Swain, Department of Chemistry, Michigan State University, East Lansing, MI; and J. Asmussen, Department of Electrical and Computer Engineering, Michigan State University, East Lansing, MI

Boron-doped polycrystalline diamond thin films are an excellent electrode material for electrochemical applications including water treatment (destruction of inorganic and organic pollutants, disinfection), electrosynthesis, electroanalytics, bioelectrochemistry and electrochemical energy storage and conversion. This broad variety of potential applications requires the material to be synthesized on various substrate materials, geometries and dimensions. Therefore a tunable microwave plasma assisted chemical vapor deposition (MPACVD) reactor was used to develop coatings for disks, plates, pins and wires made from various metals (titanium, tungsten, niobium) and silicon. Process development results are presented, which address issues such as coating adhesion, mechanical stress reduction, boron concentration and uniformity, thickness uniformity, and microscopic coating material flaws (cracks, voids). In addition the paper presents information on shaping the coated materials to fabricate designed electrodes and on testing them using cyclic voltammetry.

Wednesday, May 13

4:10 p.m. CT-14 Visible Light-Active C-Doped TiO₂ Thin Films for Solar Hydrogen Generation

M. Schulz, H.Y. Lin, M. Day, and S.I. Shah, University of Delaware, Newark, DE

_Sponsored Student Presentation_

Photoelectrochemical cells (PECs) have the potential to split water to generate clean-burning hydrogen fuel, using only input power from the sun. Titanium dioxide thin films are excellent candidates for photoanodes in PECs, and substitutionally doping pure TiO₂ with nonmetals such as carbon has been shown to improve visible light response by establishing defect electronic states just above the valence band. We have synthesized a series of modified TiO₂ thin films with atomic concentration up to 4% carbon via a simple, easily controlled pulsed laser deposition technique. Film structure and composition were verified via X-ray diffraction and X-ray photoelectron spectroscopy. UV-Visible absorption spectroscopy and preliminary electrochemical characterization showed improved visible light response over undoped TiO₂.
Wednesday, May 13

4:30 p.m. CT-15 The Properties of Metal Doped TiO$_x$ Thin Films Deposited by PVD Method

M. Horakova, Technical University in Liberec, Liberec, Czech Republic; N. Martin and E. Aubry, Institut FEMTO-ST, Besancon, France; P. Spatenka, Technical University in Liberec, Liberec, Czech Republic and University of South Bohemia, Ceske Budejovice, Czech Republic; and P. Hájková, Technical University in Liberec, Liberec, Czech Republic

For several years, titanium dioxide has gained scientific interests because of its versatile properties like photocatalytic activity. Basically, the photocatalytic process is initiated by the photogeneration of electron/hole pairs in the semiconductor by photon absorption of UV-light. The addition of noble metals into a semiconductor can change its physical properties, thus the photocatalytic activity. The aim of this study is improving photocatalytic efficiency of TiO$_x$ thin films doped with noble metals. TiO$_x$ thin films were deposited on glass substrate by DC reactive magnetron co-sputtering at low temperature and in situ doped by nanoparticles of silver, gold or copper. The morphology and structure of the films were examined by SEM and XRD. The chemical composition was studied by RBS analysis. The photocatalytic activity was evaluated from decomposition speed of aqueous solution of the Orange 7, (sodium salt of sulphonated azo dye), exposed to UV light. Relations between doped and non-doped films properties, deposition parameters and photocatalytic activity is analyzed. This work was supported by MSMT, project NANOPIN 1M0577 and by GA AV CR, project No. KAN101120701.

Wednesday, May 13

4:50 p.m. L-12 Reactive Magnetron Sputtering of ZnO:Al, A Status Report


Transparent and conductive ZnO:Al films to be applied as window layers in thin film solar cells have received strong interest in the last few years. This material competes against SnO$_2$:F by AP-CVD or LP-CVD and against ZnO:B by LP-CVD. For ZnO:Al, on the other hand, the deposition technique most relevant for industrial production is PVD by large area inline magnetron sputtering. The state-of-the-art for industrial manufacturing at the present time is ceramic target sputtering using sintered target materials either from planar or rotatable sintered targets since this technique allows for excellent process stability at acceptable cost level at this time. The reactive magnetron sputtering of ZnO:Al, on the other hand, has not achieved industrial relevance for mass production up to now due to the severe demands on closed loop control. This hindered the integration in processing lines for mass production in spite the fact that superior film properties result from reactive sputtering in terms of low absorption, high conductivity and good light scattering. Furthermore, a substantial decrease of costs can be expected when cost effective metallic Zn:Al targets are used instead of ceramic ZnO:Al$_2$O$_3$ compounds. This paper gives a report on the status of reactive AC magnetron sputtering using the 60 x 100 cm$^2$ in-line coater at Fraunhofer IST. We report on the performance of ZnO:Al coatings and thin film solar cells utilizing a variety of ZnO:Al films and also on the latest developments on the PLC based closed loop control algorithms for fully automatic operation of the coater.
Thursday Morning
May 14, 2009
Vacuum Web Coating
Thursday, May 14

8:30 a.m. W-15 Efficacy of Flexible Moisture Barrier Films Produced Using a Roll-to-Roll Coater as Measured by the Calcium Test

S. Louch, Centre for Process Innovation, Redcar, United Kingdom; M. Hodgson, Dupont Teijin Films, Middlesbrough, United Kingdom; and S. Edge and K. Luxmore, Centre for Process Innovation, Redcar, United Kingdom

Until recently, flexible barrier films implied films for food or medical packaging with oxygen transmission rates (OTR) and water vapour transmission rates (WVTR) down to ~10⁻¹cc/m²/day and ~10⁻²g/m²/day, respectively. However, since the discovery of organic semiconductors, with their inherently flexible nature, a plastic electronics revolution is imminent. In order for this to be realised, suitable substrate materials (with gas barrier performance similar to glass) and processing technologies must be developed. For example, it is often quoted that the moisture barrier performance required of polymer based substrates for organic light emitting diode (OLED) devices is of the order 10⁻⁶g/m²/day. The work presented here will explore what this level of barrier really means and, if it is achievable using roll-to-roll vacuum coating of polymer based substrates. In addition, the calcium test, a methodology for measuring WVTR barrier performance at these low levels of moisture permeation will be discussed in some detail, and results presented.

Thursday, May 14

8:50 a.m. W-16 All-in-Vacuum Deposited Transparent Multilayer Barriers on Polymer Substrates

J. Fahlteich, Fraunhofer Institute for Electron Beam and Plasma Technology FEP, Dresden, Germany

To meet the high water vapour and oxygen permeation barrier requirements of flexible electronic devices many groups suggest multilayer stacks. We have developed a concept for roll-to-roll all-in-vacuum production of multilayer barrier stacks. It is based on the combination of reactively sputtered layers with an interlayer grown by Magnetron-PECVD. Magnetron-PECVD is a novel technology that allows the deposition of both silicon-oxide like and polymer like layers using a dual magnetron system. The combination of reactive sputtering and Magnetron-PECVD has been installed in a pilot-scale roll-to-roll coating unit. Process parameters like long term stability of both processes and their combination are characterized as well as coating uniformity. Also the single and multilayer permeation barriers are characterized regarding their barrier properties against water vapour and oxygen permeation. The multilayer concept also allows the deposition of multifunctional layer stacks. An example of a combination of barrier layers and TCOs will be shown.
Thursday, May 14

9:10 a.m. W-17 Study of High Gas Barrier Performance of Film with Coated SiOxNy Layers

H. Yanagihara, Mitsubishi Plastics, Nagahama, Japan; C. Ookawara and S. Yoshida, Mitsubishi Plastics, Inc., Ibaraki, Japan; and K. Ohdaira and H. Matsumura, Japan Advanced Institute of Science and Technology, Ishikawa, Japan

Recently, a film with high gas-barrier performance for penetration of oxygen and water vapor has been strongly expected in the food packaging and the coating of electronic devices. In order to get high gas barrier performance, we have tried to coat SiOxNy layers on film by catalytic CVD (Cat-CVD). Cat-CVD is a method to make thin film by decomposing gas molecules on heated surface of catalyzer using catalytic cracking reactions and transporting them to cooled substrates. In this study, the water vapor transmission rate (WVTR) of SiNx/SiOxNy double-layer coated film showed one order superior to that of monolayer coated samples with same thickness. We will show some WVTR results mainly of samples produced by Cat-CVD, and also show measurements and other approach.

Thursday, May 14

9:30 a.m. W-18 High Rate Roll-to-Roll Deposition of ALD Thin Films on Flexible Substrates

E. Dickey and W. Barrow, Lotus Applied Technology LLC, Hillsboro, OR

Thin, transparent, dielectric barrier films were deposited on plastic web in a roll-to-roll configuration using a novel approach to Atomic Layer Deposition (ALD). The films were deposited in a roll-to-roll coater prototype in which web is transported multiple times through different precursor zones separated by slit valves. Pulsing of precursors as used in conventional ALD processes was eliminated. This resulted in very high deposition rates by eliminating the pulse and purge times. Furthermore, since different precursors were isolated from each other at all times except for the monolayer chemisorbed on the web, there was no film deposition anywhere except on the web itself. Water Vapor Transmission Rate (WVTR) data are provided showing barrier film performance as a function of film thickness for titanium dioxide films on PET. The prototype tool is described. Process parameters, including web translation speed, deposition temperature and pressure are provided. Based on these data, it is demonstrated that this technique can deposit roll-to-roll films utilizing an ALD process at extremely high rates.
Thursday, May 14

9:50 a.m. W-19 Innovative Transparent Barrier for Packaging

S. Guenther, S. Straach, and N. Schiller, Fraunhofer Institute for Electron Beam and Plasma Technology FEP, Dresden, Germany

The trend towards transparent barrier coatings for flexible packaging gains momentum. Product visibility is a powerful marketing tool. Vacuum coated transparent barrier film has been pushed forward during the last years mainly by electron beam evaporation technology with equipment installations in Japan and Europe. But as the investment cost for electron beam web coaters is high, and also as most metallizing companies are not familiar with this process, the number of running electron beam web coaters, compared to the number of Al metallizers using boats, is low. To use this basic technology of standard Al evaporation from boats also for the production of transparent barrier layers was a dream for many years. In the presentation, a new development will be described. Based on the combination of an innovative plasma-technology with standard Al evaporation from boats, transparent barrier coatings with outstanding barrier performance and optical clarity on BOPP, PET and PLA films have been achieved. A close cooperation between an equipment manufacturer, an R&D institute, and a BOPP film producer, all three partners, being leaders in their fields, have boosted this innovative development. An adapted production machine has been manufactured by Applied Materials and upscaling of this technology to production level was done in close cooperation between those involved. A first production machine using this process has been in operation since 2008 on a customer’s site.

Thursday, May 14

10:30 a.m. W-20 Polymer Nanofilms from a Topochemical Deposition/Polymerization Process

J. Lauterbach, Department of Chemical Engineering, University of Delaware, Newark, DE

*Invited 40 min. Talk*

Current polymer thin-film formation techniques give adequate film properties for the specific polymer systems and applications for which they were designed. However, they all have deficiencies that restrict their use in applications beyond those for which they were developed. Our deposition/polymerization (DP) process is a two step method to create polymer nanofilms through topochemical polymerization. First, monomer is dosed onto a sample at low temperatures in vacuum. UV radiation is then used to initiate polymerization. Using this process, the polymer films have controllable thickness in the nanometer region. As a result of the monomer being dosed onto a surface at low temperatures, the diffusion of monomer in the film is restricted. This inability of the monomer to diffuse allows for construction of complex monomer film structures, such as stacked layers. The monomers also interact when dosed as a mixture. The ability to create such mixtures allows for the use of photoinitiators and the creation of copolymers. One important characteristic of the DP process is the ability to polymerize molecules previously thought unpolymerizable. Normally, 1,2-disubstituted ethylenes are unable to polymerize because the rate of radical termination is much faster than radical propagation. In the DP process, however, high molecular weight polymers were produced.
Thursday, May 14

11:10 a.m. W-21 Plasma Enhanced Chemical Vapor Deposition (PECVD) on Web Using Novel Linear, High Density Plasma Source

M.A. George, H. Chandra, P. Morse, L. Birch, and J. Madocks, General Plasma, Inc., Tucson, AZ

General Plasma has invented a novel source technology that enables large area plasma enhanced chemical vapor deposition (PECVD) for continuous processes such as web coating. The novel source has advantages over conventional PECVD, such as no electrode coating, high precursor dissociation and no powder formation. The PBSTM deposition rates are also admirable; for examples SiO$_2$ at $> 1000$ nm-m/min and SiN at $> 200$ nm-m/min.

This paper reviews the historical progress of the new technology and presents the latest results. Films deposited by the source include hard coatings on plastics (SiO$_2$), barrier films (SiC, SiN), transparent conducting oxide (SnO$_2$), anti-reflection layers (ZnO, FeO, SiN, TiO$_2$) and active layer passivation in solar cells (SiN). Property and characterization results for these films are discussed.

Thursday, May 14

11:30 a.m. W-22 Chemistry of Powder Formation in SiO$_x$ Deposition Plasmas

M. Ricci, J.L. Dorier, and Ch. Hollenstein, École Polytechnique Fédérale de Lausanne (EPFL); and P. Fayet, Tetra Pak (Suisse) SA, Romont, Switzerland

The plasma enhanced chemical vapor deposition (PECVD) process for coating polymer films has several advantages over evaporation processes. A disadvantage of PECVD is the relatively low deposition rate which limits the line speed. The deposition rate could be increased by reducing powder formation and by an optimized use of the monomer while keeping excellent barrier characteristics. In particular, the powder formation in SiO$_x$ deposition plasmas is far from being understood compared to other PECVD processes. The plasma chemistry during powder formation has been investigated in a small capacitively coupled RF reactor equipped with advanced plasma and powder diagnostics. In particular time resolved in situ Fourier Transform Infrared Absorption Spectroscopy has been applied to elucidate the formation of the nanometer sized particles. Several different monomers for SiO$_x$ deposition, such as HMDSO, HMDSN and TMS, with various amount of oxygen admixture, have been studied. In a first phase, the chemical nature of the organosilicon compound is important, since the nucleation of the particle is induced by polymerization of the highly reactive fragments of the monomer in the plasma. In a second phase the particle size increases due to SiO$_x$ accretion which depends strongly on the oxygen content in the plasma. These results indicate the importance of the chemistry of the monomer in the nucleation phase of the particle in these reactive plasmas.
The use of optical density is key for many products in many industries. It is one of the primary measurement techniques for the determination of how thick the aluminum is on the substrate. While we in industry always hope that our Optical Density (OD) sensor never breaks, the reality is that everything will eventually fail, including your optical density sensor. While repairing a key instrument is always a priority, it may take several days depending on the type or extent of the damage. In this case, it may not be economically feasible to shutdown the machine until the sensor is repaired. How, though, can one be fairly assured to the aluminum thickness on the web over time without a sensor? The purpose of this presentation is to demonstrate how one can effectively forego the use of an OD sensor for a period of time. This presentation will go through the regression analysis by investigating the metrics chosen, the rational for choosing them, the regression models explored, and the implementation of that model.
Optical Coating
Thursday, May 14

8:30 a.m. O-17 Titanium Dioxide Thin Films: Their Structure and its Effect on their Photoactivity and Photocatalytic Properties

M.-L. Kaariainen, T.O. Kaariainen, and D.C. Cameron, ASTRaL, Lappeenranta University of Technology, Mikkeli, Finland

Atomic Layer Deposition (ALD) has been used to deposit titanium dioxide thin films. A series of films with different thicknesses has been created and the film structure has been studied with X-ray diffraction and Raman spectroscopy. It has been noticed that at a reaction temperature of 350°C titanium dioxide thin film grows first as anatase but at a certain thickness continues growing as rutile. The dependence of the photoactivity and photocatalytic activity on the film structure has been investigated. Both the photoactivity and the photocatalytic activity of the films have been found to reach their peak at a certain low film thickness. The photocatalytic activity is strongly depended on the crystallinity, especially on a high anatase/rutile ratio.

Thursday, May 14

8:50 a.m. O-18 In Situ Spectroscopic Ellipsometry for Atomic Layer Deposition

W.M.M. Kessels, Eindhoven University, Eindhoven, The Netherlands

Invited 40 min. Talk

Atomic layer deposition (ALD) is emerging as a key process in semiconductor industry as well as in many other advanced technologies employing electronic materials, optical coatings, and protective layers that require control of film properties in the (sub-)nanometer scale. In this presentation, it will be demonstrated that in situ spectroscopic ellipsometry is a very valuable tool for studying ALD film growth including ultrathin film metrology and control. Applied to the cycle-wise ALD process of oxide, nitride and metal films, spectroscopic ellipsometry yields film thickness and growth-per-cycle information. It also provides insight into the self-limiting behavior of the surface (half-)reactions, substrate nucleation effects, and evolution of the microstructure and crystal phase of the films. For conducting films, the method provides in situ analysis of their electrical resistivity, electron mean free path and conduction electron density by probing the Drude absorption in the near-infrared. These and other examples of the merits of applying spectroscopic ellipsometry to ALD will be illustrated by presenting results for metal oxides (Al₂O₃, HfO₂, Ta₂O₅, amorphous and anatase TiO₂), metal nitrides (TaN, TiN) and metals (Pt, Ru).
Thursday, May 14

9:30 a.m. O-19 End-Hall Ion Sources: Improvements, Problems, Future

V.V. Zhurin, Colorado Advanced Technology LLC, Fort Collins, CO

A review of existing industrial broad beam Hall current ion sources is presented. The main parameters determining efficiency of ion sources, latest designs and improvements will be discussed. The main types of ion sources and comparisons of some existing ion sources are described. Future Hall current ion sources are discussed. New designs and high electron emission for increase of ion beam current and ion energy are suggested. Various problems accompanying ion source operation are analyzed as are the solutions for some problems: 1) Influence of unaccounted increase of additional mass flow into ion source discharge channel that can be caused by insufficient pumping or too small dimensions of vacuum chamber, or too big ion source for a vacuum chamber; insufficient pumping that develop dangerous instabilities; 2) Charge exchange effects that are important at high test facility background pressure; 3) Presence of double ionized particles and theirs impact on ion beam energy; 4) Dielectric depositions on anode during operation with reactive gases; 5) Returned sputtered particles into an ion source with development of dielectric films on discharge channel surface, substantially influencing ion source operation; 6) Influence of ion source heating on its operation; 7) Negative ions and their role in ion beam processes

Thursday, May 14

9:50 a.m. O-20 Anisotropic Nonlinear Optical Absorption in Gold Nanorod Nanocomposite Coatings: Theory and Experiment

J.-M. Lamarre, École de Polytechnique de Montréal, Montréal, Canada; F. Billard, Institut Carnot de Bourgogne, Dijon Cedex, France; C. Harkati Kerboua, Université de Montréal, Montréal, Canada; M. Lequime, Institut Fresnel, Marseille Cedex, France; S. Roorda, Université de Montréal, Montréal, Canada; and L. Martinu, École de Polytechnique de Montréal, Montréal, Canada

Gold nanorods were formed in a silica matrix using a 3-step fabrication process: 1) hybrid deposition using plasma-enhanced chemical vapor deposition of SiO₂ and pulsed-DC co-sputtering of gold, 2) high temperature annealing, and 3) 30 MeV Cu⁺ heavy ion irradiation. Anisotropic linear and nonlinear optical properties were measured by several techniques (polarized light transmission measurement, ellipsometry and polarization-dependent Z-Scan/P-Scan) and related to the presence of transversal and longitudinal surface plasmon resonances corresponding to the particle short and long axes, respectively. Linear optical properties were modeled using the generalized Maxwell-Garnett equation and were shown to depend strongly on the nanostructure and the fabrication parameters (ion irradiation doses and annealing temperature). Gold nanorod/silica nanocomposites were found to exhibit anisotropic saturable absorption. Gold nanorod nanocomposite nonlinear absorption coefficient (at 532 nm) was found to vary between -0.9x10⁻² cm/W for a polarization along the particle short-axis and -5.1x10⁻² cm²/W for a polarization along the particle long-axis. These values were compared with reference measurements on gold nanospheres-containing samples. The anisotropic nonlinear optical properties were modeled using anisotropic local field calculations based on the Maxwell-Garnett formalism. Synthesis, microstructural control and potential applications will be discussed.
Concentrated solar power technologies (CSP) are an alternative to large area photovoltaic systems. To achieve competitive efficiency, CSP relies on highly reflective mirrors to concentrate solar radiation over a wide spectral range, from the UV at 350 nm to the near infrared at 900 to 1500 nm, with a specular reflectivity above 95%. Further requirements are - weatherability in terms of wide range humidity and temperature changes as well as durability against UV exposure, both over a design-lifetime of several decades, and economic production capabilities for large scale applications. Several options are known for fabricating high reflective mirrors. The most common approach is silver metallization, possibly enhanced by interference stacks. Additional corrosion protection layer(s) may be necessary, but need very careful selection with respect to their optical impact. The paper will discuss and compare various such coating stacks with respect to their optical performance, weatherability and capability for industrial mass production. Substrate materials are different 3D shaped polymer parts.
Thursday, May 14
11:10 a.m. O-25 ALD (Atomic Layer Deposition) for Optical Coatings
J. Maula, T. Alasaarela, and S. Sneck, Beneq Oy, Vantaa, Finland
This paper describes the use of ALD for optical coatings from the practical point of view. Features not available with the PVD are covered, like ALD capabilities to make new film materials and to modify materials during the deposition. Coating of parts in a stacked batch and/or in large areas and novel methods to get high uniformity in large batch applications is explained with examples. The possibility to use many materials in the process and capabilities to modify film interfaces for process integration purposes is explained. Factors mentioned above and its natural 3D coating capabilities, set ALD into an enabling technology position and this paper highlights these new possibilities.

Thursday, May 14
11:30 a.m. O-24 Optical Properties and Microstructure of Oxide Sculptured Thin Films by Glancing Angle Deposition
X. Xiao, Y. Jin, H. He, H. Qi, Z. Fan, and J. Shao, Shanghai Institute of Optics and Fine Mechanics, Shanghai, China
Oxide sculptured thin films were prepared by electron beam glancing angle deposition. XRD, SEM, UV-Vis-NIR spectra are employed to characterize the microstructure and optical properties. Sculptured thin film with tilted columns has optical anisotropy. Among the oxide thin films prepared, the maximum of birefringence ($\Delta n$) is up to 0.067 for TiO$_2$ at $\alpha=70^\circ$. Deposition angle and packing density are both important factors to influence in-plane birefringence. After heat treatment, the birefringence is improved greatly. The phase retardance is up to 90° at $\lambda=550$nm. Glancing angle deposition is a useful method to obtain larger in-plane birefringence, which seems useful to create some devices such as retardation plate and polarizer.
Cleantech Symposium
**Thursday, May 14**

**8:30 a.m. CT-16 An Update on the Center for Advanced Molecular Photovoltaics**

M. McGehee, Department of Materials Science and Engineering, Stanford University, Stanford, CA

*Invited 40 min. Talk*

Molecular photovoltaic cells can be fabricated at low-cost using roll-to-roll coating processes similar to those used to make newspapers. They can be much cheaper than conventional cells because, in addition to having low materials costs, the cells can be printed and connected to each other in a high-throughput, integrated architecture. Today’s best organic solar cells have an efficiency of 6.5 % and last approximately 1 year under sunlight. The Center for Advanced Molecular Photovoltaics has plans for raising the efficiency and making the cells stable for ten years or more. Highlights of recent research will be presented.

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**Thursday, May 14**

**9:10 a.m. CT-17 New Materials for Organic Photovoltaic Devices**

M.E. Thompson, M.D. Perez, K. Mutolo, and C. Schlenker, Department of Chemistry, University of Southern California, Los Angeles, CA

We have begun a systematic study of a range of different materials for organic photovoltaic devices. This talk discusses the development of new materials as donors, acceptors and buffer layers in OPVs. We have investigated a family of Ru(beta-diketonate) complexes. We have investigated a range of Ru(acetylacetonate), as a buffer material, several sulphhalocyanine complexes have been used to investigate the connection between the OPV open circuit voltage (Voc) and have explored the use of metal porphyrin complexes as donor materials in OPVs. The complexes we have chosen have high luminance efficiencies and long lifetimes. We expected that these two parameters would lead to long exciton diffusion lengths. Both Pt and Pd complexes make good red absorbing donor materials in OPVs. Unfortunately, our initial hypothesis that these materials would give long exciton diffusion lengths was wrong. We believe that this is due to a combination of both poor spectral overlap and a self quenching process that severely shortens the exciton lifetimes in thin films of these materials.
Thursday, May 14

9:30 a.m. CT-18 Advanced Processing of New Materials for Extended Operational Lifetime of Polymer Solar Cells

S. Gevorgyan and F. Krebs, Risø National Laboratory for Sustainable Energy, Technical University of Denmark, Roskilde, Denmark

The current performance in the state-of-the-art polymer solar cell is most often reached through processing methods post-film formation i.e. using solvent or thermal annealing. We present new fully roll-to-roll compatible processing methods and demonstrate that they can be used for improving the power conversion efficiency. Power conversion efficiencies in excess of 1.5% by processing novel materials in these manners will be presented. The operational lifetime is improved through choice of device geometry and materials such that exceptional resilience towards oxygen and water is achieved enabling operation without encapsulation.

Thursday, May 14

9:50 a.m. CT-19 Stability Investigations of Organic Photovoltaic Cells

J.A. Hauch, A. Seemann, F.R. Kogler, and P. Schilinsky, Konarka Technologies GmbH, Nuremberg, Germany

Stability of OPV cells is one of the key properties needed for commercialization. Despite some recent work showing that organic solar cells made with P3HT:PCBM have very promising lifetimes under various conditions, there still is not much known about the degradation mechanisms of organic solar cells. We use a novel cell architecture to investigate the influence of water and oxygen on cells in operation, allowing us to separate the degradation effects. Our results can be used to further develop packaging technology for organic solar cells, and to further improve their lifetimes.
Thursday, May 14

10:30 a.m. CT-20 Modification of Nanomorphology in Polymer: Fullerene Blends-Route Towards High Efficiency Polymer Solar Cells

Y. Yang, Department of Materials Science and Engineering, University of California Los Angeles, Los Angeles, CA; G. Li and V. Shrotiya, Solarmer Energy Inc., El Monte, CA; and Y. Yao, H.Y. Chen, and S. Sista, Department of Materials Science and Engineering, University of California Los Angeles, Los Angeles, CA

Invited 40 min. Talk

Extensive research is going into polymer solar cells because of their advantage of ease of fabrication, low cost, ability to make large area devices and mechanical flexibility. Optimizing the morphology on a molecular level is essential for achieving high performance polymer solar cells. Optimal nanomorphology leads to optimized exciton dissociation efficiency which is determined by the donor/acceptor interface area and optimal charge transport efficiency through percolating pathways for carriers. In this presentation, we discuss three strategies to manipulate the nanomorphology of regioregular poly(3-hexylthiophene) (rr-P3HT): [6,6]phenyl C61-butyric acid methyl ester (PCBM) blends viz. thermal annealing, solvent annealing and mixed solvent approach, all of which lead to improved photovoltaic performance. It has been shown that all three strategies lead to better ordering of the P3HT domains and hence good polymer crystallinity. The effect of these strategies on the blend morphology, polymer crystallinity, absorption and charge carrier mobility will be discussed and will be related to the photovoltaic performance of the device.

Thursday, May 14

11:10 a.m. CT-21 Supramolecular Approaches for Polymer-Based Solar Cells

B.C. Thompson, University of Southern California, Los Angeles, CA

Composite solar cells based on conjugated polymer donors and fullerene acceptors represent the current state-of-the-art in organic photovoltaics, where blends of poly(3-hexylthiophene) (P3HT) and [6,6]-phenyl C61-butyric acid methyl ester (PCBM) exhibit efficiencies approaching 5%. Despite the excellent performance of these systems, there is a great deal of room for improvement in not only the electronic properties of the polymer, but also in the thermal stability of the composite material. Without a precise control over donor-acceptor interfaces within the composite material, little progress can be made in improving solar cell performance. Here, several novel approaches toward polymer based solar cells will be described in which control over the supramolecular organization of the donor and acceptor components is targeted as a primary mechanism of improving the electronic properties of the solar cells. Further, steps toward achieving stable composite materials based on supramolecular approaches will be described. The ultimate goal of this work is to develop stable donor-acceptor composites in which the morphology is accurately controlled to maximize device performance and lifetime and to increase the potential for generating effective, low cost, solution processable, and flexible solar cells.
Thursday, May 14

11:30 a.m. CT-22 Alternative Hole Transport Layers for Organic Photovoltaics

D.C. Olson, J.J. Berry, N.E. Widjonarko, M.S. White, M.O. Reese, and D.S. Ginley, National Renewable Energy Laboratory, Golden, CO

Improvements in the lifetime and efficiency of organic photovoltaics are critical to its development as a viable technology. One of the important interfaces in OPV devices is between the transparent electrode and the organic active material, where an interfacial material can aid in charge extraction. In our typical OPV devices we employ a glass/indium tin oxide (ITO) substrate with an interstitial layer of PEDOT:PSS to modulate the interface between the ITO and active P3HT:PCBM bulk heterojunction (BHJ) region. The PEDOT:PSS material serves as the hole transport layer (HTL) in the devices, but has numerous drawbacks with respect to morphology, chemistry, stability and scalability. Here we report on our use of alternative HTLs based on mixed metal oxides and commercially available organic HTLs. We compare the results from P3HT:PCBM BHJ devices using these alternative HTLs with traditional devices employing PEDOT:PSS. Changes in device efficiency, as well as work function, pH, and conductivities are quantified as a function of the HTL employed. In addition, we will employ transparent electrodes other than ITO that are not accessible when using PEDOT:PSS as the HTL. We also report on initial device degradation studies, which examine the correlation between the HTL used and time dependent changes in device efficiency.

Thursday, May 14

11:50 a.m. CT-23 Large Scale Demonstrations of Polymer Solar Cells as a Tool for Innovation, Cost Analysis and Market Development

T. Nielsen and F. Krebs, Risø National Laboratory for Sustainable Energy, Technical University of Denmark, Roskilde, Denmark

Polymer solar cells have a long history approaching two decades and commercialization is imminent. There has been a large interest in establishing a firm comparison with existing solar cells technologies under conditions of large scale production and real life demonstration. In this presentation, examples of large public demonstrations of polymer solar cells produced industrially on a large scale will be detailed. The performance was significantly below the current state-of-the-art for laboratory cells but sufficient for demonstration purposes. The total process time for complete modules comprising five solution processed layers and module areas of ~100 square centimeters was as low as 12 seconds. This has allowed for extensive innovation that will be detailed along with a firm cost analysis of the technology. The market possibilities for the technology as it can be produced via the methods presented are detailed.