Monday Afternoon, November 4, 2002

Biomaterials
Room: C-201 - Session BI+VT-MoA

Protein Surface Interactions
Moderator: D. Grainger, Colorado State University

2:00pm **BI+VT-MoA1 Self-Assembled Monolayers of Carboxy-Terminated Poly(ethylene glycol): Protein Resistance, Biospecific Functionalization and Application to Immunodiagnostics**, ***R. Dahint***, University of Heidelberg, Germany **INVITED**

The high specifity of antigen/antibody reactions has been widely exploited to develop accurate detection methods for biomolecules. Heterogeneous immunoassays, where proteins are selectively bound by immobilized antibodies and detected by the use of labeled secondary antibodies are a standard diagnostic technique. Also, a considerable amount of research has been focused on immunosensor development. A general problem in immunodiagnostics is non-specific protein adsorption: Macromolecules are not only bound to the substrate by specific antigen/antibody recognition, but also adhere due to non-specific interaction forces. Hereby, the accurate determination of antigen concentration may be significantly deteriorated. Moreover, non-specifically adsorbed proteins may even block and deactivate the immobilized receptors. The integration of specific receptors into a protein resistant matrix would, therefore, significantly improve quantitative analysis. Self-assembled monolayers (SAMs) of poly- and oligo(ethylene glycol) have proven to effectively prevent protein adsorption. We, therefore, synthesized a carboxy-terminated poly(ethylene glycol) alkanethiol ($HOOC\text{-}CH_2\text{-}(OCH_2\text{-}CH_2)_n\text{-}O\text{-}(CH_2)_{11}\text{-}SH$, n = 22-45) which facilitates covalent coupling of antibodies. In contrast to most other previous studies, where receptors have been coupled to SAMs formed from a binary mixture of differently functionalized molecules, only a single chemical functionality is involved. After characterizing the films by infrared absorption (FTIR) and X-ray photoelectron spectroscopy, ellipsometry and contact angle measurements, their performance as bioselective coatings with reduced non-specific adsorption has been tested in both FTIR and acoustic wave sensor experiments. The protein resistant properties of the films are put in context with previous results on oligo(ethylene glycol) alkanethiolate SAMs including neutron reflectivity studies on protein/surface interactions.

2:40pm **BI+VT-MoA3 ToF-SIMS and XPS Analysis of Enzymatic Digests of Adsorbed Protein Films**, ***M.S. Wagner***, *D.G. Castner*, University of Washington

Characterization of multicomponent adsorbed protein films is critical in understanding biological interactions with surfaces. We have previously shown that Time of Flight Secondary Ion Mass Spectrometry (ToF-SIMS) can quantify the composition of binary and ternary adsorbed protein films using the low mass ($0 < m/z < 200$) fragmentation pattern of the mass spectrum. However, quantification of more complex protein films using this method is limited to the most abundant proteins present in the films. Therefore, trypsin digestion of the adsorbed proteins was performed to generate tryptic fragments for subsequent analysis by ToF-SIMS. The tryptic fragments were identified by combining ToF-SIMS with matrices from Matrix Assisted Laser Desorption and Ionization Mass Spectrometry (MALDI-MS). Residual protein remaining on the substrate after digestion was detected using ToF-SIMS and X-ray Photoelectron Spectroscopy (XPS). This method provides unique fragments for the identification of adsorbed proteins by ToF-SIMS.

3:00pm **BI+VT-MoA4 Protein Adsorption on Colloidal Oxide Particles**, ***K. Rezwan***, *L.P. Meier, M. Textor, L.J. Gauckler*, ETH Zurich, Switzerland

Protein adsorption to surfaces of medical implants is an essential aspect of the cascade of biological reactions taking place at the interface between synthetic material and biological environment. The types and amounts of adsorbed proteins mediate subsequent adhesion, proliferation and differentiation of cells as well as deposition of mineral phases. Most metallic biomaterials are covered by a protective, stable oxide film such as titanium oxide on titanium. Hence proteins only interact with the oxide film and not with the underlying metal. Closer investigations of the protein - oxide interface are therefore of great relevance to the biomaterials field. In the past, protein adsorption and desorption has been investigated mostly on planar surfaces by in situ techniques such as ellipsometry, optical waveguide lightmode spectroscopy (OWLS) and quartz crystal microbalance (QCM). The drawback of these methods is the lack of direct information about surface charges, which are known to strongly affect protein adhesion and conformation at interfaces. We used colloid chemistry analysis methods such as colloidal vibration potential (CVP), X - ray disc centrifuge (XDC) measurements and UV â€“ spectroscopy (UVS) to study in detail the adsorption of proteins to well-defined colloidal particles of typically 100 - 200 nm diameter. Combining these methods, the adsorbed amount of proteins and its influence on the zetapotential and the isoelectric point of the particles were determined with great precision and across a wide pH range. Adsorption of bovine serum albumin was found to alter the zetapotential of the oxides Al2O3, TiO2 and SiO2 and their isoelectric points to an extent that depended on the adsorbed mass. Combining UVS and XDC, the volume density and the thickness of the protein layer could be determined. The thickness corresponded to a monolayer or less. The adsorbed mass of albumin turned out to be nearly independent of pH in the range from pH 2 to 12.

3:20pm **BI+VT-MoA5 Prediction of Adsorption Behavior of Fibronectin as a Function of Surface Functionality Using a Customized Protein Adsorption Force-Field**, ***R.A. Latour***, *K.A. Wilson*, Clemson University, *A.J. Garcia*, Georgia Institute of Technology, *S.J. Stuart*, Clemson University

The ability of a cell to bind to an adsorbed protein layer on a biomaterial surface is dependent on the structure and availability of the protein's cell binding domains following adsorption. A well-known example of this is integrin binding to the PHSRN and RGD sites located on the 9th & 10th type III repeats of fibronectin (Fn). The objective of this research was to utilize computational chemistry to predict the relative orientation and accessibility of these cell-binding domains in Fn after adsorption as a function of surface functionality (CH3, OH, NH3+, COO-). Modeling was conducted using an SGI O2/Onyx computational system with InsightII software (Accelrys). The Charmm force-field was used to simulate intramolecular interactions for the fibronectin, while a new set of force-field parameters was created to simulate the interactions between the fibronectin and the surface. The new force-field parameters were set to provide similar energy vs. surface separation plots for peptide residue-surface adsorption as determined by previous semi-empirical modeling studies using MOPAC/PM3/COSMO. Initial energy vs Fn orientation maps were generated followed by 50 ps molecular dynamics simulations at selected positions to assess initial adsorbed Fn behavior. Results suggest that the CH3 and COO- surfaces should most strongly inhibit integrin binding, but by different mechanisms; the CH3 surface by disrupting Fn structure and the COO- surface by blocking accessibility. The OH and NH3 surfaces were predicted to preserve binding site structure and accessibility. Results compare favorably with experimental studies and provide likely molecular mechanisms that help explain experimentally observed behavior.

3:40pm **BI+VT-MoA6 Analysis of Organic and Biological Materials in Ultra-High Vacuum**, ***D.G. Castner***, University of Washington **INVITED**

Ultra-high vacuum (UHV) surface science has a long, successful history in the fields of catalysis and microelectronics. The early adaptation of UHV-based tools in these fields was largely due to the fact that the materials involved (metals, ceramics, semiconductors, etc.) were readily vacuum compatible. This talk will address the challenges of adapting UHV surface analysis techniques for analyzing organic and biological materials. These include their higher vapor pressure, their increased susceptibility to X-ray, electron, and ion sample degradation, and vacuum induced changes in their structure. Some of the first organic surface analysis experiments were done on polymers. Since that time, these experiments have been extended to self-assembled monolayers (SAMs), biomaterials, and adsorbed biomolecules. Examples to be discussed from these areas will include the effect of polymer additives, surface rearrangement of polymers, the well-defined structure of SAMs, and preserving the conformation of adsorbed proteins.

4:20pm **BI+VT-MoA8 PEG-ylated Surfaces with Graded Protein Interactiveness : A ToF-SIMS, XPS and Optical Waveguide Sensor Study**, ***S. Pasche***, *S.M. De Paul, J. Vörös*, Swiss Federal Institute of Technology, *P. Hug, B. Keller*, Swiss Federal Laboratory for Material Testing and Research, *H.J. Griesser*, University of South Australia, *N.D. Spencer, M. Textor*, Swiss Federal Institute of Technology

Poly(L-lysine) grafted with poly(ethylene glycol) (PLL-g-PEG), a polycationic co-polymer positively charged at neutral pH, has been shown to spontaneously adsorb onto negatively charged surfaces, rendering them protein-resistant to a degree related to the PEG surface density. Since the PEG surface density is a function of polymer architecture (PEG molecular weight and grafting ratio expressed as number of lysine monomers per PEG side chain), it becomes feasible to control the interactiveness of a surface by varying the co-polymer architecture. Angle-dependent XPS and ToF-SIMS

were used to investigate the surface-chemical properties. The adsorbed mass after serum exposure was determined by an optical sensor technique. Further colloid-modified AFM force measurements aim at studying the mechanical properties of the coated surfaces. PLL-g-PEG was adsorbed onto niobium oxide coated wafers, resulting in the formation of stable polymeric monolayers. The grafting ratio, g, of the polymer was varied systematically between 2 and 10, leading, upon surface adsorption, to highly different, but controlled PEG surface densities. PEG molecular weight was varied between 1000 and 5000. Polymer adsorbed mass was determined quantitatively by an in situ optical waveguide technique. A quantitative relationship was established between EG-monomer surface density, calculated from the known polymer architecture and the surface-adsorbed mass, ToF-SIMS intensities of PEG-, PLL- and substrate-related secondary ion peaks, and the amount of serum proteins that adsorbed onto the different polymer-coated surfaces. PLL-g-PEG surface-coating technology allows the fabrication of surfaces with tailored interactiveness and the establishment of design criteria for PEG-based, protein-resistant surfaces.

4:40pm **BI+VT-MoA9 Time-of-Flight Secondary Ion Mass Spectrometry Analysis of Conformational Changes in Adsorbed Protein Films**, ***N. Xia***, University of Washington, *C.J. May*, Yale University, *S.L. McArthur*, *D.G. Castner*, University of Washington

Characterizing the identity, composition, conformation, and orientation of adsorbed proteins is essential for the development of biocompatible devices. Static time-of-flight secondary mass spectrometry (ToF-SIMS) is a powerful surface analytical technique for analyzing adsorbed protein films. However, the ToF-SIMS experiment is done under vacuum, and drying adsorbed proteins for analysis can denature or change their conformation. In this study, trehalose coating was used to inhibit these conformational changes from occurring during sample preparation for ToF-SIMS analysis. Surface plasmon resonance (SPR) analysis showed that air-dried films of trehalose-stabilized antibodies retained a significant proportion of their hydrated antigen binding activity. In contrast, air-drying without trehalose protection resulted in the adsorbed protein films losing most of their antigen binding activity. Structural differences between trehalose-stabilized and unstabilized protein films were then analyzed with static ToF-SIMS. By application of principle component analysis (PCA) to the ToF-SIMS spectra, the biological activity difference observed in SPR was correlated to changes in protein conformation. Trehalose-protected proteins retained a greater degree of their original conformation than the unprotected proteins. This suggests that static ToF-SIMS has the capability to distinguish conformational differences in adsorbed protein films. Moreover, trehalose protection can be used for static ToF-SIMS analysis of adsorbed protein films to obtain structural information that is more relevant to the structure of the proteins in aqueous conditions.

5:00pm **BI+VT-MoA10 Study of the Adsorption Kinetics and Conformational Changes of Human Serum Albumin and Human Plasma Fibronectin using PM-RAIRS, Radiolabelling and Atomic Force Microscopy**, ***R.J. Manning***, *C.M.J. Fauroux*, *M.J. Pilling*, *P. Gardner*, *G.J. Leggett*, University of Manchester Institute of Science and Technology, UK

The kinetics of adsorption of proteins has been studied on self assembled monolayers (SAMs) on gold, formed by the adsorption of alkanethiolates with differing functional groups and varying alkyl chain lengths. The adsorption of human serum albumin (HSA) and human plasma fibronectin (HPF) has been studied using three complementary techniques: post modulation fourier transform reflection adsorption infrared spectroscopy (PM-RAIRS), radiolabelling, and atomic force microscopy (AFM). Initial adsorption kinetics of HSA and HPF were established using FTIR. It was found that monolayer coverage was reached faster on methyl terminated SAMs than on hydroxyl and carboxylic acid terminated hydrophilic monolayers. Tritium radiolabelling of HSA and HPF confirmed the trends observed with FTIR. The conformations of the adsorbed proteins were followed using PM-RAIRS, enabling quantitative monitoring of the percentage of α-helix, β-sheet, β-turn and random coils, indicating the degree of denaturation on differing surfaces over time. Finally, AFM was used to generate direct observations of layers of adsorbed proteins, providing useful insights into the distribution of proteins across the differing surfaces and enabling individual molecules to be observed. HSA was found to form a fibrillar network on methyl terminated SAMs at low concentrations and short adsorption times, whilst individual molecules were observed on hydroxyl and carboxylic acid terminated monolayers. This study demonstrates the complimentarity of FTIR, radiolabelling and AFM in understanding the adsorption of proteins on well-ordered SAMs.

Tuesday Morning, November 5, 2002

Vacuum Technology
Room: C-104 - Session VT-TuM

Novel Vacuum Materials and Pumps, Including Getters
Moderator: M.L. Ferris, SAES Getters USA

8:20am **VT-TuM1 Expanded Characteristics Evaluation for Low Vacuum Dry Pumps**, ***J.Y. Lim***, *S.H. Chung, W.S. Cheung, K.H. Chung, Y.H. Shin, S.S. Hong,* Korea Research Institute of Standards and Science, *W.G. Sim,* Hannam University, Korea

Since positive-displacement dry pumps were first launched into the semiconductor industry in 1984, issues concerning about characteristics evaluation on consistent bases have been continuously arisen from the mass production lines. Besides their clean and continuous pumping capability, occasional devastating malfunctions or characteristic degradations of such pumps during the manufacturing processes have been also reported in the Korea semiconductor industry. On behalf of these issues, the integrated characteristics evaluation system for dry vacuum pumps has been developed collaborating with several semiconductor and branch dry pump manufacturers in Korea. The evaluation system exploits the constant volume flowmeter to measure the mass flow rate in standards level, and facilitates the evaluation of spatially averaged sound power levels using a reverberation chamber.[1] New and overhauled roots, claw, classical screw, and scroll type pumps supplied from the manufacturers have been evaluated using the evaluation system in terms of ultimate pressure, pumping speed, vibration, and sound power. The correlation analyzed among those results shows clear signs of pump degradation related each other. We selected the mass flow measuring method with a constant chamber volume of 874 L because of its direct monitoring capability not allowing blind mass flow rate measurement, and proved the method allows us to measure five decades of mass flow rates from 0.01 to 10^3 mbar-l/s with an uncertainty of ±3% which is within the internationally accepted standards limit. In this work the integrated characteristics evaluation method has been significant because of pump degradation or malfunction symptom to be further understood and predicted.

[1]W.S. Cheung, J.Y. Lim, K.H. Chung, Experimental study on noise characteristics of dry pumps, The 2002 International Congress and Exposition on Noise Control Engineering, Inter-noise 2002, Dearborn, MI, USA.

8:40am **VT-TuM2 High Throughput Continuous Cryopump, with Gas Dynamic Compression of the Helium Minority Stream, for Pumping Fusion Reactors**, ***C.A. Foster***, Cryogenic Applications F, Inc., *S. Willms, S. Letzring,* Los Alamos National Laboratory, *D. Schechter*, Cryogenic Applications F, Inc.

An analysis of the flow of gases in a large cryopumping system designed to pump a magnetic plasma fusion reactor is presented. The pumping system for a 3gigawatt thermal reactor must handle a throughput of 2 Pa-m3/s of He and 200Pa-m3/s of D/T and maintain a pressure in the diverter at about 0.5Pa. A cryopump which removes the cryo-ice during operation with a regenerating head or "snail" is capable of pumping the D/T stream. A set of twelve 500mm bore pumps would each pump 16.7 Pa-m3/s of the D/T stream with the He being pumped by a set of turbo pumps downstream to the cryopumps. In designing the entrance baffle and analyzing the gas flow in the pump, it was determined that whereas the gases were close to free molecular flow conditions in a room temperature design, at 30K they would be in the viscous flow regime. The viscous flow conductance of the entrance duct at room temperature was not drastically different from that calculated using the free molecular flow equations. However, Poiseuille flow has a strong temperature dependance, so that the pressure drop across a pipe in viscous flow at 30 K is dramatically lower than at 300K. Since a cold baffle is typically used to precool the gases entering a cryopump, it was decided to replace it and the room temperature ducts with a refrigerated duct operating in the viscous flow regime. An analysis of long cold ducts replacing the conventional vacuum pipes between the reactor diverter chamber and the cryopumps allowed the pipe size to be reduced by a factor of four in area. This is especially advantageous in a reactor since the vacuum ducts have to pass between the coils and through the neutron shielding blankets. An analysis of the fluid flow of the D/T and He in the cold ducts showed a gas dynamic drag compression of the He minority species(10X to 30X) by the D/T stream as was utilized in the Gaede diffusion pump. This pre-compression allows conventional turbo-molecular pumps to be used as the compound helium pumps.

9:00am **VT-TuM3 Vacuum System Design and Simulation Program for PCs**, ***R.A. Langley***, *L.R. Baylor, P. LaMarche,* Oak Ridge Scientific Consultants

A new integrated software package for Windows has been developed to design vacuum systems and to simulate existing vacuum systems. The package is modeled after earlier DOS based programs by Santeler.[1,2] The initial setup of the design calculation allows the choice of up to five parallel pumping modules on the vacuum chamber with the choice of up to six different gas types. At this stage, the primary pump for each module is chosen. Each pumping module is then separately addressed to choose the optimum secondary pump or pumps for that pumping module. Both circular and rectangular cross section tubing is allowed. Pumping speed data for many types of pumps and many sizes of pumps is maintained within the program and additional pumping speed data can also be input for use by the program. The calculation is based on a precision set of gas flow equations for all pressure conditions, i.e. molecular, transition, and viscous, and provides pumpdown and steady state data. True gas fractionation is calculated and gas flow for tubes of varying lengths, i.e. from an orifice to a long tube, can be calculated. Various examples of the use of the program will be presented.

[1] Donald J. Santeler, Vacuum System Design, Donald J. Santeler Assoc.
[2] Donald J. Santeler, VSD-II Vacuum System Design, Donald J. Santeler Assoc.

9:20am **VT-TuM4 Design Fabrication and Processing of Vacuum Chambers for High Energy Accelerators at Brookhaven***, ***H.C. Hseuh***, *M. Mapes, D. Weiss*, Brookhaven National Laboratory **INVITED**

There are several distinct accelerators and storage rings at Brookhaven ranging in length from tens of meters for the Tandem to several kilometers for the Relativistic Heavy Ion Collider. The vacuum systems of these facilities must provide a suitable environment for the circulating beams while also being subjected to the intense bombardment of various energetic particles. The unique physics requirements and material selection criterions for the vacuum chambers will be described. The fabrication, cleaning and conditioning of the vacuum chambers will be reviewed. The treatment, sealing techniques and performance of non-conventional materials such as ceramic and ferrites in an ultrahigh vacuum and high radiation environment will also be presented. *Work performed under the auspices of the U.S. Department of Energy.

10:00am **VT-TuM6 Advanced Materials and Fabrication Techniques for the Next Generation Light Source**, ***J.R. Noonan***, *G.A. Goeppner, J. Gagliano, R.A. Rosenberg,* Argonne National Laboratory, *D.R. Walters*, Veeco International **INVITED**

The next generation of light source will probably be based on electron linear accelerators using a laser driven photocathode gun. The accelerator requirement will be very stringent: very short pulses (< 100 fs, >1,000 amp peak current, and large electric accelerating gradients > 30 MeV/m). These specifications will impose new, significant requirements on the vacuum systems. For example, the cathode material in the photocathode gun not only must have high photo-electron yields, but also must withstand high laser power and high RF electric gradients. The beam tube must be fabricated to new tolerances with respect to surface finish, surface resistance, and change of cross section. The new accelerator requirements are leading the need for new materials and manufacturing technology. The talk will review research at several Free Electron Laser accelerators, and how the technology assisted the success in VUV photon emission from free electron lasers.

10:40am **VT-TuM8 Investigations of Novel Getter Materials**, ***W. Knapp***, *D. Schleussner*, Otto-von-Guericke-Universität Magdeburg, Germany, *T. Stenitzer*, Konstantin Technologies GmbH, Austria, *K. Chuntonov*, Alkali Metals Ltd., Israel

The novel chemisorbents on the basis of alkali, alkali-earth and rare-earth metals have recently emerged as one of the most promising getter materials. The novel getters are based on an A-B alloy system, where A is a chemically active metal and B is a fusible non-volatile metal. Metal B is creating a protective shell on the whole surface of the novel getter material, which is impermeable during handling and storing and permeable when activated. The main advantages of the novel getters compared to the standard getter materials in use are: - Much higher kinetic and capacity sorption, - Significant lower activation temperatures, - Very small size and flexible design possible, - Easy handling of very active materials, e.g. Li, Na, Cs etc. In quantitatively proof of advantages and for basic getter investigations an ultra-high vacuum (UHV) experimental setup with a high measurement standard was developed. Getter temperatures and gas flow

rates are regulated for different operations. On the basis of precise total and partial vacuum pressure measurements the getter sorption capacities are valued and compared in dependence on vacuum pressure range and kind of gases. For this with our experimental setup different measurement methods are possible, like pressure rise, throughput and difference method. We investigated sorption capacities of standard and novel getters using the pressure rise method. In our presentation comparative results of our investigations are presented and discussed.

11:00am **VT-TuM9 Modern Types of Getters for Novel Applications**, ***L. Rosai***, Saes Getters, Italy

Modern applications of gettering aimed to remove unwanted impurities and contaminants in vacuum and gas filled environments encompass new types of evacuated or gas filled devices, each requiring a tailor made solution in terms of gettering materials, geometry, process integration. In addition to the traditional types of getters in form of rings, porous pellets and coated strips, nowadays new configurations are available to meet the specific requirements of new applications: the getters can be prepared in form of very porous thin films sheets, coated in- situ as sputtered films, patterned on a semiconductor substrate, made in form of thin permeable bags filled with different materials each devoted to a specific gettering task, filling small capsules equipped with special mounting features. In these modern getters the so called activation process is no longer necessary thanks to special features of getter packaging and/or a natural activation occurring during device manufacturing. New types of getter pumps, not requiring substitution for many months or years are also available, for different applications in portable instruments, semiconductor, LCD and magnetic recording media manufacturing In some of these applications these new getter types are necessary not only for ensuring a long life to the device but also as a process aid during the manufacturing of the device itself .The vacuum and purity requirements of the most recent applications of getters will be illustrated. These will include: Vacuum Insulated Panels, Vacuum Pipes, Flywheels, Plasma Display Panels , Field Emission Displays, OLEDs, MEMS, Opto-electronic devices, Magnetic recording devices, Semiconductor and LCD production tools.

11:20am **VT-TuM10 Gaede Langmuir Award Address: The Impact of Non-evaporable Getters on the Evolution of UHV/XHV Technology**, ***C. Benvenuti****, CERN, Switzerland **INVITED**

Non-Evaporable Getters (NEG) entered the field of UHV technology with the advent of powder-coated gettering strips. Coated strips are particularly well suited to provide large, linear pumping for the conductance limited vacuum chambers of particle accelerators. An example of this application is the pumping system of the Large Electron Positron collider (LEP) at CERN. The development of alloys of lower activation temperature (about 400 °C) may be seen as another major break-through in the NEG technology. In this case the activation may be achieved "passively" during the bakeout of a stainless steel vacuum system, resulting not only in a simplified solution (no need of electrical insulation, feedthroughs and power supply), but also in improved vacuum performance (larger pumping speed and capacity, lower ultimate pressure). The final stage of the NEG technology evolution has been reached with the recent development of thin getter films coated on the inner surfaces of a vacuum chamber. These films offer an even lower temperature of activation (180 °C), a feature which renders them applicable also to copper and aluminum structures. In addition, they provide large and evenly distributed pumping, suppress the thermal degassing of the underlying material and strongly reduce, after activation, both the degassing and the secondary electron yield induced by the surface bombardment. The impact of NEGs on the evolution of vacuum technology will be illustrated and the new possibilities offered by the thin film coatings will be reviewed with the help of some examples.

***** Gaede Langmuir Award Winner

Tuesday Afternoon, November 5, 2002

Vacuum Technology
Room: C-104 - Session VT-TuA

Vacuum System Architecture and Specialized Analytical Techniques
Moderator: J.L. Provo, Sandia National Laboratories

2:00pm **VT-TuA1 Present Status of the KEKB Vacuum System**, *K. Kanazawa, S. Kato,* ***Y. Suetsugu****, H. Hisamatsu, M. Shimamoto, M. Shirai*, High Energy Accelerator Research Organization (KEK), Japan

The KEK B-Factory (KEKB) is an electron-positron collider with an asymmetric energies consisting of two rings, that is, the High Energy Ring (HER) for 8.0 GeV electrons and the Low Energy Ring (LER) for 3.5 GeV positrons. The design beam currents are 1.1 A and 2.6 A with 5120 bunches for HER and LER, respectively. Each ring has a circumference of 3016 m and most of beam chambers are made of oxygen free copper for its ability to withstand the intense heat load and to shield effectively the radiation from high beam currents. The pumping scheme is a combination of Non-Evaporable Getter (NEG) pumps and sputter ion pumps. The commissioning of KEKB started in December 1998. At the end of March 2002, the achieved stored currents were about 0. 93 A and 1.44 A for HER and LER, respectively, with 1200 bunches. The KEKB is now able to serve the world-record luminosity of 7.2 nb^{-1} s^{-1} for the BELLE detector. The vacuum system has been operating satisfactorily. The average pressure of about $1x10^{-7}$ Pa is achieved now for both rings during the operation. The coefficient of the photon stimulated gas desorption (PSD) decreased steadily to almost $1x10^{-6}$ molecules $photon^{-1}$ at the integrated linear photon flux of about $3x10^{25}$ photons m^{-1}. Most of vacuum components, such as Helicoflex sealing (Le Carbone Co. Ltd.) and the vacuum bellows with RF-shield structure, have been working well. The severest trouble had been the beam-induced troubles of the movable masks, but the newly developed masks were installed and are now in use without serious problem. One of the latest issues is the excess heating of bellows, chambers or pumps coming from the electromagnetic filed excited by the intense bunched beam. A non-linear dependence of pressure on beam current has been observed in LER. That seems to deeply relate to the electron multipactoring, which causes the electron cloud instability. Here we will summarize the experiences and the present status of KEKB vacuum system, and touch briefly the future plan.

2:20pm **VT-TuA2 Development of Sputtering System for Large-Area Deposition of $CuIn_{1-x}Ga_xSe_{1-y}S_y$ Thin-Film Solar Cells**, ***N.G. Dhere***, *A.H. Jahagirdar, A.A. Kadam, V.S. Gade, H.P. Patil*, University of Central Florida

Manufacturing cost of $CuIn_{1-x}Ga_xSe_{1-y}S_y$ (CIGS) thin-film modules is expected to become cheaper than that of crystalline silicon modules within 5 years. At present, commissioning and reaching full production of thin film modules is delayed because of the non-availability of turnkey manufacturing plants. Few university laboratories are conducting research on design and construction of PV plants. CIGS thin-film solar cells are being prepared routinely at FSEC on glass and metallic foil substrates for terrestrial and space applications. Earlier the size was limited to 3x3 cm^2. This paper presents results of development efforts in design and construction of large-area sputtering system for large-area (15x15 cm^2) CIGS thin-film solar cells. The system has the potential of serving as a nucleus of a pilot plant for fabrication of CIGS minimodules. It could be used for simulating full-scale production set-up. Stepper-motor controlled, linear substrate movement and uniform argon distribution set-ups were designed and built. RF tuning network was modified for optimum impedance matching. Initial problems of bowing of the backing diaphragm, possible formation of air pockets, restriction of effective water flow and consequent heating of the target material were resolved by increasing the thickness of the backing plate and redesigning the structural members. Thickness distribution was optimized by modifying the magnetic field distribution in the middle 15-cm portion of the 10x30 cm^2 magnetron sputtering sources by selectively removing nickel-coated soft-iron pieces at the rear. The present optimum configuration has resulted in thickness variation of ±3% over 11.5x10 cm^2 for Mo, CuGa, In, ZnO, and ZnO:Al layers. Magnetic field is being boosted at extremities to avoid precipitous ~15% drop beyond 11.5 cm and to achieve thickness uniformity of better than ±2% over 12.7x12.7 cm^2 and ±3% over 15.3x15.3 cm^2 areas.

2:40pm **VT-TuA3 Ultra-sensitive Detection of Helium Release from Metal Tritides**, ***J. Poths***, *T.J. Venhaus, F.J. Steinkruger*, Los Alamos National Laboratory **INVITED**

Static noble gas mass spectrometry has been in use for ultra-sensitive analysis of samples in the geological community for almost 5 decades. We have been using a magnetic sector instrument created by Al Nier at the University of Minnesota in both geological and nuclear research. In static mode of operation, the sample is equilibrated into the volume of the instrument with no pumping, rather than flowing through the source and into a pump as in a standard dynamic mass spectrometry. Thus, static operation provides a factor of 10,000 increase in sensitivity. We have recently applied this technique to analyzing the release of helium-3 from erbium ditritide films. The increased sensitivity has allowed us to compare helium release on timescales ranging from 5 minutes to 100 days. We confirm that helium release is highest at the beginning of an erbium ditritide film's lifetime, then decreases after a few months to a steady rate. Interestingly, during this period of steady-state release, the helium release rate seems to be independent of the buildup of helium-3 levels in the film. This observation suggests that at the moment of tritium decay a helium-3 atom is either immediately released or is trapped and no longer accessible for release. At steady-state, the ratio of helium released to helium decayed during storage is about 0.008.

3:20pm **VT-TuA5 Quantification of Mass Spectra in Experiments with Deuterium**, ***B. Zajec***, *V. Nemanic, M. Zumer*, Institute of Surface Engineering and Optoelectronics, Slovenia

For monitoring the interaction of gaseous hydrogen with surfaces or bulk materials, deuterium is frequently applied as a tracer gas for the quadrupole mass spectrometer (QMS) analysis. In fast and steady processes, the major peak at mass number 4 is frequently a good indicator of deuterium involved reactions while peaks at mass numbers 2 and 3 may be neglected. In slow and transient processes, the weak peaks at mass numbers 2, 3 and 4 must be considered for a quantitative analysis. Tracing reactions where deuterium molecules dissociate is troublesome since the mass number 2 overlaps with the hydrogen background. The problem to eliminate the QMS artefacts is usually solved by calibration of the instrument with a suitable low deuterium flux, but any further resolution of the background hydrogen originated from the chamber surfaces is very inaccurate. We present the results of a calibration procedure of two QMS mounted on a well outgassed UHV system, realised in a specific way. Each of the QMS was pumped in line but could be also separated from the system by a valve. A constant deuterium or hydrogen inflow was set from 10^{-8} mbar l s^{-1} to 10^{-6} mbar l s^{-1} by observing the pressure rise in the chamber by means of a calibrated capacitance manometer. The calibration of both QMS was taken in the dynamic mode for both gases. Further on, the spectra of deuterium accumulated in the UHV system for a defined period of time were compared to the calibration spectra. From the difference, we could resolve the extent of mass numbers 2 and 3 produced in the QMS from the contribution of the deuterium participated in surface reactions at the chamber wall. It was thus shown that after admittance of pure deuterium into the UHV chamber at 10^{-4} mbar, its exchange with the adsorbed hydrogen led in some hours to a noticeable changed proportion of mass numbers 2, 3 and 4. This could not be predicted from the low background outgassing rate.

3:40pm **VT-TuA6 Mass Spectrometric Determination of Hydrogen and Hydrogen Isotopes from Thin Films**, ***J.F. Browning***, Sandia National Laboratories

The absolute measurement of hydrogen and hydrogen isotope concentrations in materials is of interest to many areas of both applied and fundamental research. In this work we describe a technique for the high accuracy determination of hydrogen concentration in thin films. The technique involves the thermal desorption of hydrogen from the film at a temperature of 900°C. Application of the ideal gas law to such experiments is questionable at best due to the extreme temperature range existing between the thermal desorption system and the mass spectrometer inlet system. To overcome issues associated with such extremes in temperature we use a response function technique to quantitate the total molar quantity of gas evolved from the film followed by high resolution mass spectrometric determination of the constituent components. Molar concentrations in the range 10^{-8} to 10^{-5} are routinely determined to an overall uncertainty of ±1%.

4:00pm **VT-TuA7 Quantification of Gas Load from Ultra-small GDP Capsules**, ***S.D. Balsley***, Sandia National Laboratories

A-priori knowledge of the gas composition of 10^{-8} liter capsules manufactured by General Atomics Corporation (GA) and used in inertial confinement fusion (ICF) experiments at Sandia National Laboratories' Z-pinch facility is important to experimentalists and modelers. These Z-pinch driven capsules are larger than any ever fielded by any other ICF program, and as such are part of an ongoing research program at GA and Sandia. Total pressure determinations via burst tests, although useful, lack information regarding the composition of gas mixtures. Here we present a novel technique for quantitatively determining partial pressures of gas loaded capsules whereby a low-background vacuum fixture for capsule bursting is coupled with a Finnigan-MAT 271 mass spectrometer. Typically, capsules consist of a 50µm thick polymer coated with a 3µm polyvinyl alcohol layer. Capsules are diffusion filled with a deuterium-argon mixture that is pre-defined by model calculations. Nominal fill pressures for deuterium-argon capsules are 17atm and 0.075atm, respectively. Other gas fills are also used, including deuterated methane (CD_4) mixed with minor quantities of argon or tetramethylsilane. Initial test results from several argon filled capsules agree well with stated fill pressures. Subsequent analysis of sister capsules similar to those imploded at Z-pinch in 2002 show good correlation between stated fill quantities and expected deuterium loss due to diffusion. It is envisioned that application of this technique will be important for gas fill validation of target capsules to be used in the National Ignition Facility, scheduled to begin testing in 2003-2004.

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

4:20pm **VT-TuA8 How to Have Clean Surfaces in an Unclean World**, ***R. Robinson***, *D.D. Allred, A. Guillermo, R. Sandberg, A Jackson,* Brigham Young University, *M.K. Newey,* University of Maryland

Clean surfaces that are exposed to the atmosphere rapidly become dirty. Even though a surface may be carefully prepared and well characterized in its preparation or deposition environment, there is no guarantee that it will remain unchanged minutes after it is exposed to the air. We will report our measurements on the amount of contamination a sample acquires from various activities including handling with various kinds of gloves, sitting face up or down in the lab for several hours, storage in wafer carriers etc. We will also discuss the use of several cleaning techniques: UV light+ozone, plasma ashing, and polymer (Opticlean) for removing dust and/or the contamination layers in preparing materials for EUV/VUV (8-110 eV) and/or AFM measurements. Experience from silicon-based microelectronics is instructive but not definitive here. Answers depend in part on the identity of the surface. XPS shows the presence of carbon and oxygen in most contamination layers. Spectroscopic ellipsometry's advantages include: it can be used in air, measurements are rapid and it is sensitive to the relative thickness of layers even in the sub nm range. Measurement is the first step to understanding which can lead to control. We are making materials for VUV and EUV (8-100 eV) optical applications where the presence of a few nanometers of carbon and/or oxygen containing materials can have a remarkable effect on the performance of the surface. A companion group is doing AFM. Here the presence of organic contamination can clog the tip. We will report our answers to questions such as: How fast does a surface acquire its accidental carbon-containing overcoat? What can be done to block or ameliorate deterioration? Can the sample be handled, cut and stored for several hours before the next measurements? How can a sample be restored or recleaned without losing its essential characteristics? When does storage introduce its own problems?

Vacuum Technology
Room: Exhibit Hall B2 - Session VT-TuP

Poster Session

VT-TuP1 The KATRIN Neutrino Mass Experiment - Vacuum Technological Aspects, ***C. Day***, *V. Hauer*, Forschungszentrum Karlsruhe, Germany, *J. Bonn*, University of Mainz, Germany

In modern particle physics, one of the most challenging task is to determine the rest mass of neutrinos. The energy spectrum of the electrons of the β decay can be used to derive upper limits of the electron neutrino mass. A new large tritium experiment is currently being planned, the KATRIN experiment, which is expected to increase the resolution of mass determination by one order of magnitude. It is an international effort and will be built up and operated in FZK, Germany. The KATRIN vacuum system can be subdivided into three main parts, the windowless gaseous tritium source (WGTS), the differential pumping section, and the pre- and main spectrometer. The WGTS introduces tritium gas into the central piping system, operated under fine vacuum conditions. Further in spectrometer direction follows a differentially pumped section to reduce the gas pressure by nine orders of magnitude. This is realised by a cascade of turbomolecular pumps combined with a cryogenic pump system (Ar frost). Between the tritium source and the main spectrometer a pre-spectrometer is inserted, acting as an energy pre-filter. The key component of the new experiment is the large MAC-E-filter with a diameter of 7 m and an overall length of about 20 m, designed for XHV conditions. The spectrometers are pumped by a two-stage system, comprising turbomolecular pumps with high compression ratio for hydrogen, and getter pump modules. This paper delineates the underlying concepts for the three different vacuum systems. Special requirements are full tritium compatibility, operation in strong magnetic fields and under high voltage conditions. The differential pumping section, especially the final cryogenic pump, must provide a capture probability of almost unity, to allow for XHV conditions in the pre-spectrometer tank. To limit the pumping speed requirements with respect to the getter pumps, the outgassing of the spectrometer vessel walls must be reduced to an absolute minimum.

VT-TuP2 Extended Measurements of Photon Stimulated Desorption from a Copper Beam Chamber after Removal of Surface Oxide[1], ***C.L. Foerster***, *C. Lanni*, Brookhaven National Laboratory

Photon Stimulated Desorption(PSD) was measured from a copper beam chamber after completely removing the vacuum surface oxide in order to reduce the PSD. Continuous measurements have been recorded for the chamber, over a year and a half exposure period, to determine long term exposure effects. The measurements of PSD and specular photon reflection were performed on NSLS beamline U9a at Brookhaven National Laboratory. It is well known that PSD causes a pressure rise in accelerator and storage ring vacuum, which limits their performance. For this experiment, a KEKB factory beam chamber from a previous experiment was chemically etched and chemically cleaned prior to installation on beamline U9a. Previous PSD measurements have shown that this chemical treatment removes any memory of prior exposure or conditioning. After installation, the copper chamber and end stop were vacuum baked to 250°C for more than a week to completely remove vacuum surface oxides. The chamber was exposed to more than $5x10^{24}$ photons direct from the source having a critical energy of 595 eV and striking at an incident angle of 100 mrad. The major PSD yields for hydrogen, carbon monoxide, carbon dioxide, and methane are reported as a function of accumulated photon flux and preparation. The PSD yields for the copper chamber, after oxide removal, were found to be greatly reduced when compared to previous measurements at this laboratory and by those reported from other laboratories. The PSD component gases remained the same during the long exposure and all were significantly reduced. Carbon dioxide and methane were reduced much more than hydrogen and carbon monoxide. Specular photon reflection did not change significantly during the extended exposure.

[1]Work performed under the auspices of the U.S. Department of Energy, under contract DE-AC02-98CH10886.

VT-TuP3 Synchrotron Radiation-Induced Desorption of NEG-Coated Vacuum Chambers at the ESRF, ***R Kersevan***, European Synchrotron Radiation Facility, France

The outgassing yield of several vacuum chambers under exposure to synchrotron radiation at the European Synchrotron Radiation Facility (ESRF) is given. Recently, Non-Evaporable Getter (NEG) coatings have been applied in order to reduce the pressure profile inside narrow-gap vacuum chambers, with the aim of reducing the interaction between the 6 GeV electron beam and the residual gas. Chambers made out of stainless steel, with copper coating, and extruded aluminum have been studied. Wherever possible, comparison between un-coated and NEG-coated chambers of the same geometry is made. It is shown that the NEG-coated chambers give a reduced amount of bremsstrahlung radiation, a clear indication of a reduced outgassing yield and distributed pumping. A brief description of a NEG-coating facility, capable of coating chambers up to 6 meters in length, being built and commissioned at the ESRF is given.

VT-TuP4 New Absorber in a Ceramic Kicker-chamber for the TLS Electron Storage Ring, ***G.-Y. Hsiung***, *S.-N. Hsu, C.-S. Ho, J.-R. Chen*, Synchrotron Radiation Research Center, Taiwan

An insertion device of 6T super-conducting wiggler has been installed in the injection section of the Taiwan Light Source (TLS) electron storage ring. A ceramic kicker-chamber downstream the device subjects an intensive synchrotron radiation on both side-wall. A water-cooled copper absorber, installed inside the ceramic chamber to shield the inner wall from irradiation, might cause serious problems. The problems contain the induced arcing between the absorber and the ceramic chamber, the induced interference magnetic field, etc. during the ignition of the kicker magnet. The efficiency of beam injection and the beam life time could be seriously degraded. The solution to cure those problems, the design of the absorber, and the commissioning results will be described in this paper.

VT-TuP5 Numerical Simulation of the Ion Beams Transmission Efficiency For the Design of the DC-72 Cyclotron Vacuum System, ***A.V. Tikhomirov***, *G.G. Gulbekian, R.Ts. Oganessian*, Joint Institute for Nuclear Research, Russia

The results of transmission efficiency numerical simulation for the ion beams due to a charge exchange with the residual gas in the cyclotron vacuum system including: the axial injection system; the cyclotron vacuum chamber as well as transport lines for accelerated ion beams are presented. Simulation method have been developed and tested on the base of experiments at four heavy ion cyclotrons of FLNR. Simulation results have provided determination of the main parameters for the DC-72 cyclotron vacuum system.

VT-TuP6 Study of the Performance of a Precision Constant Volume Flowmeter for Vacuum Calibration and Measurement, ***Y.W. Chang***, *J.S. Lin*, Precision Instrument Development Center, Taiwan, R. O. C.

The Vacuum Laboratory at Precision Instrument Development Center in Taiwan has developed a high vacuum calibration and measurement system by the orifice flow method. A constant volume flowmeter is used to provide a measurable, steady and uniform gas flow to the vacuum chamber in the range 10^{-2} to 10^{-5} Torr.l/s. With this type of flowmeter gas is supplied from a reservoir of fixed and known volume. The pressure drop in the volume is measured and the product of reservoir volume and the pressure drop per unit time determines the gas throughput of flowmeter. A regulating valve at the exit of reservoir controls the gas flow rate. The performance of such a flowmeter is verified by introducing the known gas flow rate to an orifice calibration chamber to generate a pressure, which can be determined by the flow rate and the orifice conductance under molecular flow conditions. The predicted pressure is compared with the pressures simultaneously measured by a spinning rotor gauge calibrated at PTB in German and an ion gauge calibrated at NML in Taiwan. When the correction factors for two gauges show the same tendency in repeated measures, the variations of correction factors then indicate the performance of the flowmeter. The pressure drops in the reservoir are measured both absolutely and differentially. In absolute way the reservoir pressure is measured with a capacitance diaphragm gauge (CDG); while in differential way the pressure difference between the reservoir and a reference volume is measured with a differential CDG. The measurement results of gas throughput of the flowmeter by two ways are also presented in this study.

VT-TuP7 Influence of Temperature on the Sensitivity Coefficient of a Hot Cathode Ionization Gauge, ***H. Akimichi***, *M. Hirata*, National Institute of Advanced Industrial Science and Technology, Japan

Hot cathode ionization gauges, such as a triode gauge and a Bayard-Alpert type gauge are used as reference, secondary and transfer standards in high and ultrahigh vacuum. For these applications, the stability of the sensitivity coefficient (S) of the gauge is very important. Under ideal conditions, the coefficient may be kept within 1%. It is well known, however, that the indication of the gauge is strongly affected by temperature. We studied the influence of temperature on the indication of the gauge. By cooling an ionization gauge with an electric fan, the indication at pressure lower than

about 10^{-4} Pa decreased with temperature, which dues to the decrease in the outgassing from the gauge itself. Typically, the indicated pressure at $6x10^{-6}$ Pa decreases about 30 % by the decrease in the temperature (Tg) of the gauge head from 40 to 27 °C. In contrast, at pressure higher than about 10^{-4} Pa, the decrease in the temperature caused an increment of the indicated pressure at about 2 %. The shift in the indicated pressure was explained by the thermal transpiration effect $S'/S = \sqrt{(Tg/Tg')}$. Change in room temperature (Tr) from 22 to 32 °C, on the other hand, the sensitivity coefficient of the gauge changed from 0.128 to 0.125 Pa^{-1}. This result was also explained by the relations $S'/S = Tr/Tr'$, since the temperature change in vacuum chamber and in gauge head are much the same. These results mean that compensation of the temperature for the sensitivity coefficient is important for a precious pressure measurement using an ionization gauge.

VT-TuP8 Influence of Gas-Surface Interaction on Thermal Transpiration of a Capacitance Diaphragm Gauge, *S. Nishizawa, H. Akimichi,* ***M. Hirata***, National Institute of Advanced Industrial Science and Technology, Japan

For a capacitance diaphragm gauge (CDG), the temperature difference between the sensor head and the vacuum chamber gives a non-linear sensitivity, which is called thermal transpiration. This sensitivity depends on gas species and pressure. It is supposed that under the same condition of gas-surface interaction, the sensitivity should be normalized by mean free path regardless of gas species. However, the sensitivity dependence of mean free path is also different from gas species. For example, at molecular flow regime, the sensitivity of He is slightly small in comparison with Ar and N_2. It means that the gas-surface interaction should be different from each gas. In this study, by using a direct simulation Monte Carlo (DSMC) method, the influence of gas-surface interaction on thermal transpiration was analyzed. In case of random and cosign reflection models, the sensitivity has non-linearity and depends on mean free path. On the other hand, in case of a perfectly elastic reflection model, the sensitivity is constant regardless mean free path. In case of complex reflection that is composed of random and elastic reflections, as increasing the elastic reflection component, the sensitivity decreases from that of random and cosign reflection to elastic reflection. From these results, it is suggested that the elastic reflection component of He-surface interaction is larger than Ar and N_2-surface interaction. It means that as decreasing the molecule diameter, the elastic reflection becomes important.

VT-TuP9 Vacuum Chamber with Distributed Titanium Sublimation Pumping for the G-Line Wiggler at Cornell High Energy Synchrotron Source, ***Y. Li***, *Y. He, N.B. Mistry,* Cornell University

This paper describes a 3-meter long vacuum chamber for the newly installed wiggler magnet at the Cornell Electron Storage Ring (CESR) for the synchrotron light beam line of the Cornell High Energy Synchrotron Source (CHESS). Copper was chosen as the main chamber material for its good electric and thermal conductivities. Proper mechanical design and welding procedure were implemented to meet very tight tolerances to ensure adequate vertical aperture for the stored beams in CESR while allowing the required small wiggler gap. Distributed titanium sublimation pumping is incorporated along 3-meter length of the chamber to provide sufficient pumping speed and capacity for CESR and CHESS operations. The chamber pumping performance was evaluated prior to the installation. Linear distributed pumping speeds at the beam line of ~800 liter/sec/meter for N_2 and CO and ~4200 liter/sec/meter for H_2 were measured. The pumping speed is determined by the gas conductance of the slotted copper screen between the beam line and the TiSP compartments. The measured pumping capacities for N_2, CO and H_2 are ~1.0, ~2.0 and ~77 torr-liter, respectively, for each titanium sublimation cycle. Measurements also showed that CO molecules adsorb on the N_2 and H_2 saturated titanium films with virtually the same initial sticking coefficient as on a fresh titanium film. Detail analyses indicated very different CO adsorption mechanisms between the N_2 and H_2 saturated titanium films. While the replacement of surface H_2 by CO was observed, little desorption of N_2 was measured. Operational experience showed excellent vacuum pumping performance over seven months after the chamber installation.

VT-TuP10 XPS Studies of Al and Cu Samples Exposed to an Accelerator Environment, ***R.A. Rosenberg***, *M.W. McDowell, Q. Ma,* Argonne National Laboratory

Designers of present and future particle accelerators are becoming increasing concerned about the influence of the components surface chemistry on the accelerator performance. Bombardment of these surfaces by photons can cause desorption of gases and prod uction of primary and secondary electrons. In some cases interaction of these electrons with the particle beam and the chamber walls can lead to an amplification of the electron density which in turn can cause degradation of the beam. It is well known th a t exposure to an accelerator environment can cause "conditioning" of the chamber surfaces. In order to understand the manner in which the surface structure might influence the production of gases and electrons in the accelerator it is necessary to stud y s uch surfaces both before and after exposure to accelerator conditions. There have been numerous studies performed on representative materials prior to being inserted into an accelerator but very little done on materials that have "lived" in the accele r ato r for extended periods. In the present work we mounted Al and Cu coupons at different positions in a section of the Advanced Photon Source storage ring and removed them following exposures ranging from 6 to 18 months. XPS surface analysis was perfor me d be fore and after exposure. Changes were observed that depended on the location and whether the coupon was facing the chamber interior or chamber wall. These results will be presented and compared to data obtained from laboratory measurements meant to simulate the accelerator conditions. Work supported by U.S. Department of Energy, Office of Basic Energy Sciences under Contract No. W-31-109-ENG-38.

VT-TuP11 A Low Cost Method to Deposit Diamond Films on WC-Co and Si Substrates, ***S. Nasrazadani***, University of North Texas

Diamond Coatings are known to be highly desirable for their wear resistance as well as high thermal conductivities. High hardness, low coefficient friction makes diamond attractive for mechanical properties while, high thermal conductivity make it suitable for substrate material as a heat sink. Low cost deposition of this material was investigated using hot filament chemical vapor deposition. Effects of chamber gas composition was evaluated in morphology of diamond films formed on WC-Co and Si substrates.

VT-TuP12 Method to Control the Amount of Helium Delivered during Leak Testing, ***F.E. Juravic Jr.***, Fermilab

Purpose The purpose of this paper is to demonstrate a method for limiting the amount of helium administered during leak testing and provide a method for keeping the atmospheric helium in a location to a minimum to eliminate backstreaming into the system. This method utilizes the permeability of a balloon. The transporting of helium to the leak check area is also safer by not requiring a cylinder in the leak check location. Utilizing the many shapes of balloons and partially filling of the balloon, any configuration can deliver helium to the leak location. The balloon I filled for the test fell to the floor with the amount of helium I put into the balloon.

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Situation where a high background of helium cannot be tolerated will be avoided by limiting the amount of helium brought into the room during leak checking. Transporting helium in the balloon into remote areas will allow the technician from accidentally introducing huge amounts of helium that can backstream into the msld and can be kept in the remote areas where the space in limited. This method can be applied to all forms of leak checking. This method is not recommended for certification for cryogenic systems.

Wednesday Morning, November 6, 2002

Thin Films
Room: C-101 - Session TF+VT-WeM

Atomic Layer Deposition - Barriers & Nitrides
Moderator: S.M. Rossnagel, IBM T.J. Watson Research Center

8:20am **TF+VT-WeM1 The PE-ALD of Ta Based Metals/Nitrides: The Growth, Materials Properties, and Applications to Future Device Fabrications**, ***H. Kim***, *S.M. Rossnagel*, IBM T.J. Watson Research Center **INVITED**

Thin film deposition techniques producing high quality and highly conformal films with atomic level control are increasingly required as semiconductor device size shrinks into nanoscale regime. Atomic layer deposition (ALD) is expected to play an important role in depositing thin layers in nanoscale Si device manufacturing. Plasma enhancement of the process allows deposition at significantly lower temperatures than both conventional thermal ALD and chemical vapor deposition. Among the key materials used for today's semiconductor processing, thin films of inert, refractory materials will continue to be used in interconnect applications as diffusion barriers, seed and adhesion layers as well as potential front end applications such as contacts or gate metallization. In this presentation, the Ta-based ALD systems have been explored at low temperature for a variety of semiconductor devices applications. Ta-based metals/nitrides films were grown by plasma-enhanced atomic layer deposition (PE-ALD) at temperatures from room temperature up to 400 °C using an inorganic halide source and RF plasma-produced atomic H as metal precursor and the reducing agent, respectively. The growth mechanism, microstructure, and chemical composition were studied using various ex situ analyses techniques. Good quality films with low contamination levels were obtained at low growth temperatures. Additionally, thermal stability, diffusion barrier property, resistivity, and other electrical properties, which are the essential materials properties for semiconductor device fabrication, were investigated. These results indicate that the PE-ALD process scales to manufacturing dimensions and applications and will facilitate the extension of interconnect technology beyond 100 nm dimensions.

9:00am **TF+VT-WeM3 Characteristics of TiN Films Deposited by rf Remote Plasma Enhanced Atomic Layer Deposition (ALD) Method using Metal Organic Precursor**, ***S. Seo***, *J. Kim, Y. Kim, Y.D. Kim, H. Jeon*, Hanyang University, Korea

Titanium nitride (TiN) has been most widely used as a diffusion barrier in ULSI devices because of its very low resistivity, good chemical and thermal stability, and impermeability to Si diffusion as well as the excellent adhesion to Si and SiO_2 films.[1-3] TiN barrier layer has been deposited predominantly by sputtering and chemical vapor deposition method.[4] However, as the device dimension has been shrinking down continuously, TiN films deposited by sputtering and CVD have faced the serious problems such as poor step coverage and conformality. Also, especially for the TiN films deposited by CVD using metal organic precursors, a relatively considerable amount of carbon impurity is still incorporated into the TiN films. For these reasons, we investigated TiN films deposited by rf remote plasma enhanced atomic layer deposition (ALD) technique which is expected to reduce or eliminate the problems related with sputtering and CVD.[5] TiN films were deposited using tetrakis-dimethyl-amido-titanium (TDMAT) as Ti precursor and ammonia (NH_3) and reactant gas at the optimized ALD processing windows. Rf remote plasma was used to reduce the carbon incorporation and to enhance chemical stability. The physical, chemical and electrical characteristics of TiN films were analyzed using Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), rutherford backscattering spectrometer (RBS), cross-sectional transmission electron microscope (XTEM) and four-point probe method.

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[3]M. Eizenberg, MRS Bull. 20 (1995) 38.
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[5]H. Jeon, J. W. Lee, Y. D. Kim, D. S. Kim and K. S. Yi : Vac. Sci. Technol. 18 (2000) 1595.

9:20am **TF+VT-WeM4 Importance of Hydrogen Recombination on Flow Tube Walls During Hydrogen Radical-Assisted Metal Atomic Layer Deposition**, ***R.K. Grubbs***, *S.M. George*, University of Colorado, Boulder

Many metals can be deposited with atomic layer control using hydrogen radical-assisted metal atomic layer deposition (ALD). Designing the hydrogen plasma source and hydrogen radical delivery for efficient hydrogen radical-assisted metal ALD in a viscous flow reactor offers many challenges. In particular, hydrogen recombination on the flow tube walls to form H_2 is a serious loss to the hydrogen radical flux. To quantify the hydrogen radical flux and its loss caused by hydrogen recombination on the flow tube walls, a dual thermocouple probe was constructed based on exposed and covered Pt/Rh thermocouple junctions. This probe measured hydrogen radical concentration by determining the heat evolved from hydrogen radical recombination on the exposed thermocouple surface. The thermocouple probe was then employed to measure hydrogen radical concentration versus distance from the hydrogen radical source for four flow tube materials. The hydrogen radical concentration decreased dramatically versus distance and was different for stainless steel, aluminum, pyrex and quartz flow tube materials. By modeling the decrease in hydrogen radical concentration versus distance, a hydrogen radical recombination coefficient could be determined from the data. The recombination coefficient ranged from $\gamma = 1.5 \times 10^{-4}$ for stainless steel to $\gamma = 5.7 \times 10^{-5}$ for pyrex. Given the magnitude of the hydrogen radical recombination coefficients, the reaction chamber for hydrogen radical-assisted metal ALD must be positioned very close to the hydrogen radical source.

9:40am **TF+VT-WeM5 Improved Nucleation of TiN ALD Films on Low k Polymer Dielectrics Using Al_2O_3 ALD Adhesion Layers**, ***C.A. Wilson***, *J.W. Elam, M. Schuisky, Z.A. Sechrist, S.M. George*, University of Colorado

Diffusion layers are required to prevent copper from diffusing into low k polymer dielectrics in backend interconnects. The ability to deposit conformal diffusion layers, such as TiN, onto high aspect ratio low k polymer features requires atomic layer deposition (ALD) techniques. This study examined TiN ALD on low k polymer dielectrics using tetrakis-dimethylamino titanium (TDMAT) and NH_3. X-ray fluorescence spectroscopy (XRFS), optical microscopy and surface profiling of the TiN ALD films deposited on the low k polymer dielectrics revealed discontinuous films displaying distinct patchy regions of thinner TiN coating. To study TiN ALD nucleation, in situ quartz crystal microbalance (QCM) measurements were performed by spin-coating a low k polymer dielectric onto the QCM sensor. Subsequent QCM measurements during TiN ALD revealed very low initial TiN ALD growth rates indicating poor nucleation. Al_2O_3 ALD was then performed on the low k polymer dielectric using trimethyl aluminum and H_2O. Surface profiling, XRFS, QCM and transmission electron microscopy measurements revealed that the Al_2O_3 ALD films nucleate immediately on the low k polymer dielectric producing continuous Al_2O_3 films. In addition, QCM measurements showed that TiN ALD nucleates readily on the Al_2O_3 surface. Intermediate Al_2O_3 ALD adhesion layers may facilitate the growth of continuous TiN ALD films on low k polymer dielectrics. Examination of TiN ALD films prepared on low k polymer dielectrics with progressively thinner Al_2O_3 ALD adhesion layers revealed that 10 Al_2O_3 ALD cycles are sufficient to promote the nucleation of the TiN ALD films.

10:00am **TF+VT-WeM6 Alternating Layer Deposition of Dielectric Films**, ***A.P. Paranjpe***, *B. McDougall, K.Z. Zhang, W. Vereb*, TORREX **INVITED**

Interest in alternating layer deposition (ALD) for the deposition of thin films used in semiconductor devices has grown rapidly due to the numerous advantages offered by ALD. Often, relatively high exposure doses are required to achieve self-limited surface saturation that is necessary for good uniformity, conformal deposition in high aspect ratio features, low impurity incorporation, and superior electrical properties. The low deposition rates make ALD using conventional reactors impractical in a semiconductor manufacturing environment for film thickness values > 10 nm. We describe a Parallel Wafer Processing reactor architecture capable of processing 1 - 25 wafers simultaneously that provides a 3X - 4X throughput advantage over single wafer ALD processes for both standalone and dustered operation. This reactor architecture is well-suited for the deposition of SiN and SiO_2, since initial surface chemisorption rates are relatively high, but high exposures (> 10 Torr-s) are required to achieve self-limited surface saturation. Exceptionally smooth (Ra < 0.15 nm), stoichiometric SiN films with conformality of ~ 100% in high aspect ratio features can be deposited at 0.1 - 0.2 nm/cycle in the temperature range of 450 - 550°C. The hydrogen content and wet etch rates are significantly lower than CVD SiN films deposited at equivalent temperatures confirming that ALD provides superior film quality. Electrical leakage is lower compared to SiN deposited in a conventional furnace at 750°C. SiN films have also been deposited via cyclic CVD which is analogous to ALD except that the chemisorption of the Si source deviates from the ideal self-limited behavior. Cyclic CVD

offers deposition rates that are up to 50% higher than deposition rates achievable in ALD, but film properties including conformality are intermediate between ALD and CVD films. We will also discuss the deposition of other dielectric films using the Parallel Wafer Processing reactor.

10:40am **TF+VT-WeM8 Photochemically-Assisted ALD of BN Thin Films**, ***J. Olander***, *M. Ottosson, K.M.E. Larsson*, Uppsala University, Sweden

Boron nitride-based materials have properties like high thermal stability, oxidation resistance and interesting electronic properties and are thus suitable materials for electronic devices, heat resistant semiconductors and lubricants. Thin films of BN materials have only to limited extent been prepared and the film properties are not well known. A combined experimental and theoretical investigation of BN growth from NH3 and BBr3 has been performed. Thin films of Boron Nitride have been deposited on SiO2 by means of Laser-Assisted Atomic Layer Deposition (ALD) using an ArF excimer laser. The deposition temperatures were between 300 and 750 °C. In order to investigate the growth at an atomic level, theoretical calculations have been performed using the DFT method. Theoretically, the adsorption of the NH3 and BBr3 and their fragmented components were found to be favorable for continued growth of cubic BN. However, the films obtained in the present study are of turbostratic structure. Both NH3 and BBr3 were observed to strongly absorb light from the ArF excimer laser. As a result of the photofragmentation of the precursors, the growth rate of the BN films was increased. At lower temperatures, the densities of the grown BN films were also enhanced.

Vacuum Technology
Room: C-104 - Session VT-WeM

Outgassing
Moderator: L.A. Westerberg, The Svedberg Laboratory

8:20am **VT-WeM1 Influence of Thermal and Surface Treatments on the Outgassing Of Austenitic Stainless Steels Studied by Thermal and Electron Stimulated Desorption**, *C. Benvenuti*, ***P. Chiggiato***, *G. Chuste, I. Wevers*, CERN, Switzerland **INVITED**

Austenitic stainless steel is at present the most widely used material for UHV applications. In order to improve its vacuum behavior, several post-production treatments are usually applied. Surface chemical and electrochemical processing are used to reduce surface contamination and/or roughness, while heating, either under vacuum or in air, is applied to decrease hydrogen thermal outgassing. The effect of these treatments on the content and on the binding state of surface and bulk gases has been studied by thermal desorption spectroscopy and electron stimulated desorption. The enrichment of hydrogen induced by surface electro-polishing and the effect of the residual hydrogen pressure during vacuum firing have been studied in detail.

9:00am **VT-WeM3 Comparison of Outgassing Rates of Bare and TiN-coated Stainless Steel Vacuum Chambers***, *P. He, H.C. Hseuh, M. Mapes,* ***R. Todd***, *D. Weiss, D. Wilson*, Brookhaven National Laboratory

The stainless steel vacuum chambers of the 248m accumulator ring of Spallation Neutron Source (SNS) are to be coated with ~ 100 nm of TiN to reduce the secondary electron yield. Each SNS chamber is approximately 4m long and over 20cm in diameter. The coating is produced by DC magnetron sputtering using a long cathode imbedded with permanent magnets. Reports in literature suggest that the potential benefit of a TiN coating as a hydrogen permeation barrier that reduces the ultimate outgassing rate. The outgassing rates of several SNS chambers were measured with and without TiN coating of various thicknesses, and before and after in-situ bake. No improvements in outgassing were observed with the coated chambers. The results of the coated chambers were compared with the surface structure and thickness of the TiN coatings as analyzed using AES and SEM. A correlation between film density and outgassing rate was found. *Work performed under the auspices of the U.S. Department of Energy.

9:20am **VT-WeM4 Degassing of Static Expansion Vacuum Gauge Calibration Chamber**, ***J. Setina***, Institute of Metals and Technology, Slovenia

Laboratory of Pressure Metrology at the Institute of Metals and Technology is developing a static expansion system for calibrations of vacuum gauges. The gasses evolved from the chamber walls significantly affect the accuracy of generated calibration pressure at low pressures below 1mPa. The chambers of the expansion system were designed in accordance with established ultrahigh vacuum practice and are made from stainless steel. We studied the outgassing characteristics of the large calibration chamber by the gas accumulation method. The chamber was sealed-off from the pump and the rate of pressure rise was measured using the spinning rotor gauge (SRG). From this the total outgassing rate can be determined. At the end of the accumulation period the chamber was evacuated again. During evacuation the composition of the accumulated gas was dynamically analyzed with the quadrupole mass spectrometer. The method of dynamic gas analysis will be described and outgassing of unbaked and baked chamber will be compared. Bake-out temperature was gradually increased from 60 °C to 250 °C. By bake-out at 100 °C for 50 hours we effectively removed water vapour and other adsorbed gases and the main outgassing component became H2 at a rate of 1x10-11 mbarls-1cm-2. Final outgassing rate after 160 hours of bake at 250 °C was 2.4x10-13 mbarls-1cm-2. Measured H2 outgassing rate after bake-out at 100 °C increased by a factor of 3 compared to the initial value of unbaked surface. This indicates the importance of the adsorbed layer on stainless steel surface on the kinetics of H2 evolution. The diffused hydrogen atoms from the bulk must recombine into H2 molecule in order to be de-sorbed. Other adsorbed gasses can hinder the recombination of hydrogen atoms.

9:40am **VT-WeM5 Organic Contaminants Adsorption Behavior on Silicon Wafer Surface Under Reduced Pressure**, ***T. Hayashi***, *T. Kawaguchi, N. Tanahashi,* Tohoku University, Japan, *M. Saito, K. Suzuki,* Tokyo Electron Ltd., Japan, *Y. Wakayama,* Taisei Corporation, Japan, *Y. Shirai, T. Ohmi*, Tohoku University, Japan

It is known that organic contaminants on silicon wafer surface cause many detrimental effects, such as degradation of gate oxide integrity, yield losses, and so on. To prevent that, closed manufacturing system has been proposed. However, these are considered only for outside of process chamber under atmosphere pressure. And now, the low-pressure processes such as CVD, sputter, dry etching, etc. are routinely used under several hundred mTorr or less. The organic contamination behavior on silicon wafer surface in low-pressure chamber has not been reported yet. In this study, we clarified the organic contamination behavior on silicon wafer surface under reduced pressure. We have researched the following three results; 1) The amount of organic compounds adsorbed onto a silicon wafer stored in a chamber under reduced pressure was much more than that stored under atmosphere pressure. 2) The volatile level released from pure n-Eicosane, typical hydrocarbon contaminant, does not depend on the pressure. 3) The adsorption behavior for the amount of volatile from n-Eicosane adsorbed onto silicon wafer surface coexisted with n-Eicosane in the low-pressure chamber is in accordance with Langmuir model of monolayer adsorption and the amount of adsorption saturated is in inverse proportion to pressure. In conclusion, we clarified that the inner pressure of a chamber become lower, the partial pressure of released organic compounds from chamber components becomes higher, that is, the contact frequency between organic compounds and silicon wafer surface become higher. As a result of that, the much amount of organic compounds is adsorbed onto silicon wafer surface.

10:00am **VT-WeM6 Study on Desorption of Carbonaceous Gas Molecules from Copper Surfaces under Electron Bombardment and the Surface Characterization**, ***M. Nishiwaki***, KEK, Japan, *S. Kato*, KEK & The Graduate University for Advanced Studies, Japan

In particle accelerators, the carbonaceous gas desorption is generally observed as a result of the energetic particle stimulation at ducts and r.f. component surfaces during the operation and would deteriorate the beam performance. Therefore we aim to understand origin of the desorbed carbonaceous gas from the copper surface using carbon isotope. The passive layer cannot be formed at the surface of the oxygen free copper adopted to the beam duct. Since the surface should be easily altered due to irradiation of energetic particles and/or exposure to residual gas, the carbonaceous gas desorption mechanism from the surface is of high interest. In this study, we focused on the electron stimulated desorption (ESD) from the copper surface and its in-site surface characterization by x-ray photoelectron spectroscopy and Auger electron spectroscopy. For the surface cleaning and initializing, the copper samples were sputtered with Ar^+ ion beam and annealed. Exposure of ^{13}C isotope gas and ^{13}C ion implantation were done to the samples with the known quantities respectively. Afterwards, ESD rates from the samples were measured using throughput method with a calibrated residual gas analyzer quantitatively. By using the isotope, influences of ^{12}C in the residual gas and the bulk of copper can be eliminated during observation of desorbed gas from the sample under electron bombardment. The ESD of ^{13}C related gas species from the surface exposed to $^{13}CO_2$ were not observed. In the experiments of ^{13}C implanted samples, the ESD of ^{13}C related gas species with a low current density were not observed either at the room temperature. However the ESD rates of ^{13}C related gas species from the heated ^{13}C implanted sample around 65 degrees C were as high as the thermal desorption rates at around 250 degrees C.

These results showed that the diffusion of implanted ^{13}C from the bulk was enhanced by heating and/or electron bombardment depending on the beam current density and might suggest that origin of the desorbed carbonaceous gas is not the top surface but the bulk possibly in a range of the electron penetration depth.

10:20am **VT-WeM7 Solubility and Diffusion of Hydrogen in Ordered Metals**, ***P. Repa***, *L. Peksa, T. Gronych, R. Ulman*, Charles University, Czech Republic

In the materials with ordered structure, such as the nanocrystalline and gradient materials, a relatively great part of the material is influenced by the interfaces between the crystals or between the components. As the interfaces differ from the bulk by the density and the topology of atoms, physical properties of the materials are influenced. As a result, the metals of that structure exhibit changes not only in ductility and other mechanical properties but also in the electric and magnetic properties as well when exposed to hydrogen. That's way knowledge of hydrogenation kinetics of the materials have become in the great interest recently. Since the measurement of the solubility and diffusion coefficient of hydrogen in the ordered metals is complicated due to mechanical properties of the measured samples, requirements on the experimental set up and measuring procedure is discussed. Results of the measurements are presented in which a sample was exposed to the hydrogen at elevated temperature in a filling chamber for a satisfactorily long time period to be saturated. Then the saturated sample was gradually heated up in a UHV system and the variations of the total pressure and partial pressures of several selected gases were registered. The total amount of gas dissolved in the sample, the values of solubility and diffusion constants could be estimated by an analysis of the obtained experimental data. A strong dependence of the solubility of hydrogen on the structure and composition of structured metals was discovered.

Vacuum Technology
Room: C-104 - Session VT-WeA

Vacuum Measurements, Components, and Control
Moderator: P.C. Arnold, Helix Technology, Inc.

2:00pm **VT-WeA1 Eight Unconventional Gauges: A User's Impressions**, ***B.R.F. Kendall***, Elvac Laboratories **INVITED**

Conventional vacuum gauges are unlikely to fill emerging needs for accurate measurements below the mid 10E-10 Torr range. There is also an unmet need for rugged, accurate, inexpensive sensors operating in the low micron range. The solutions may lie in the development of modern versions of gauges now found mainly in the pages of the more arcane textbooks. Laboratory evaluations are given for a number of original and updated gauges likely to be useful in modern applications. For the UHV range these include the extractor, modulated Bayard-Alpert, and X-Ray-neutralized Bayard-Alpert gauges, plus several variants of the inverted magnetron and magnetron cold-cathode gauges. For the micron range, test results are given for thermistor, viscosity and molecular drag gauges. The need for fresh approaches to vacuum gauging is particularly important at this time, when the recent trend has been to restrict commercial production to a few of the most popular general-purpose types. Formation of a users' group to encourage continued production and development of reliable UHV gauges is suggested.

2:40pm **VT-WeA3 The Effect of Ambient Temperature on the Sensitivity of Hot-Cathode Ionization Gauges**, ***P. Abbott***, National Institute of Standards and Technology, *P. Mohan*, National Physical Laboratory, India

A recent comparison of the high vacuum standards of several National Metrology Institutes (NMIs) was performed over the range of 10^{-6} to 10^{-3} Pa using hot-cathode and spinning rotor gauges as transfer standards. Among the participants, their laboratory ambient temperatures varied by as much as five degrees Celsius. It is necessary to know how laboratory temperature affects the sensitivity of the hot-cathode transfer standards (spinning rotor gauges explicitly account for the gas temperature) so that individual laboratory results can be corrected accordingly. The results are presented for an experiment in which the sensitivities of several hot-cathode ionization gauges were measured for ambient laboratory temperatures between 23 and 31 degrees Celsius. It was found that all of the ionization gauges exhibited very similar behavior, and that the sensitivity dependence on temperature could be adequately modeled with a linear fit over the investigated temperature range.

3:00pm **VT-WeA4 The Method of Calibrating and Adjusting Sensitivity of Mass Spectrometer**, ***Y. Feng***, *D.T. Li, D.X. Zhang*, Lanzhou Institute of Physics, P.R. China

The mass spectrometer is used as a partial pressure gauge in many fields on vacuum, especially in monitoring and measurement of vacuum system. It is necessary to calibrate the parameters of the mass spectrometer in order to improve the accuracy during the pressure measurement. A vacuum system, Calibration apparatus of mass spectrometer for partial pressure analysis with dynamic flow method, has been established in our center, which can be used to calibrate some parameters of the mass spectrometer with one kind of gas or some kinds of mixed gases. In this paper, the principle of the system is introduced, and the method and results for calibrating the sensitivity of QMS422 which manufactured by BALZERS is given. In the process, we get the curve on the sensitivity change with the emit ion current, the cathode volts and the focus volts change. At last, the better method is recommended to make the mass spectrometer in the good measurement condition with the largest or the most stable sensitivity.

3:20pm **VT-WeA5 Using DeviceNet for Improved Vacuum Monitoring**, ***C.E. Karlsen***, Lawrence Livermore National Laboratory **INVITED**

DeviceNet is a new standard for an industrial, device-level network for connecting a wide variety of industrial instruments, actuators, and control computers. Over 300 manufacturers make products that are certified by ODVA to work with this standard. It reduces overall installation costs by minimizing wiring and improves measurement accuracies by eliminating transmitted analog signals. This talk will give an overview of the DeviceNet standard as applied to vacuum systems. It will cover typical costs, performance issues, design parameters, and some of the products available. It will include a study on the use of DeviceNet on the Spatial Filter Vacuum System in the National Ignition Facility (NIF) at the Lawrence Livermore National Laboratory (LLNL.).

4:00pm **VT-WeA7 Closed Loop Process Control for Reactive Sputter Deposition of Dielectric Films**, ***D. Carter***, *H. Walde, G. McDonough, G.A. Roche*, Advanced Energy Industries

Pulsed-dc reactive sputter deposition of dielectric films has be an active area of study over recent years. It has been demonstrated that transition region sputtering can produce quality dielectric films at high deposition rates making this approach attractive to the alternatives of high frequency sputtering from ceramic targets. As with all processes, time based control and repeatability are critical to the acceptance of such technology. While voltage reversal during pulsing has proven effective in stabilizing arc activity, additional controls are required to stabilize the reactive environment to ensure film composition and controlled sputtering target condition. Various techniques are available to monitor the sputtering environment including partial pressure, optical emission and sputter source impedance but all typically require the addition of costly, complex, intrusive and sometimes unreliable components if one is to be used in a feedback-control-loop system. This study looks at a non-intrusive, cost effective approach to the incorporation of a Closed-Loop-Control (CLC) system in a pulsed-dc reactive sputter deposition process for the production of Al2O3 and SiO2 thin films. This approach uses target voltage as the primary feedback parameter to directly control the setpoint to a high-speed sonic piezo-driven mass flow controller. Target transition region control is demonstrated in the aluminum-oxygen and silicon-oxygen systems and the approach is evaluated for film quality, long-term process stability, process repeatability, and susceptibility to arcs and other potential anomalies encountered in such difficult to control reactive processes.

4:20pm **VT-WeA8 Soft LaserBellows**, ***D.U. Chang***, LaserTech USA

New and innovative welding technology brought precision, high quality "soft" metal bellows to practical reality. Thin gage annular disks are laser welded automatically with the aid of computer-controlled precision guidance by a machine vision seam tracking system. The resistance to the axial motion of the bellows (spring rate) is reduced to 47% to 19% compared with conventional bellows of the same size. These "soft" bellows are especially suitable for hermetic sealing of vacuum valves and manipulators for ultra-high vacuum and semi-conductor applications. Higher positioning accuracy and low motor power are some of the benefits of these bellows. The "soft" bellows are 100% checked for weld integrity by automatic welding/inspection machines, followed by mass spectrometer leak check. Randomly selected samples go through life cycle testing. The bellows last more than 5 million cycles without failure. Finite element analysis (FEA) of the bellows was used extensively in the design of bellows. Comprehensive life cycle testing and leak testing verified the analytical prediction.

4:40pm **VT-WeA9 An Ultra-sensitive Leak Detection and Calibration System**, *Ping Chen, Xu Chen, Q. Jin,* ***Liangzhen Cha***, Tsinghua University, P.R. China

Although the minimum detectable leak rates can be as low as 10^{-12}Pa*m^3/s for most commercial dry leak detectors, but demands on lower and reliable detection limit are challenging for highly reliable vacuum devices. To meet these demands, an ultra-sensitive leak detection and calibration system is developed. In order to improve the dynamic leak detection limit, it is necessary to decrease the background noise of the vacuum system. An all-metal ultra high vacuum system consisted of a turbo-molecular pump, an ion pump, a dry roughing pump and an UHV comparative Quadrupole Mass Spectrometer is developed with a minimum working pressure of 10^{-7}Pa. The leak detection limit can still be improved if the undesirable background gas is pumped out by a getter pump during the accumulation process. By using a 10^{-7}Pa*m^3/s molecular flow platinum wire-glass reference leak[1] with an adjustable device to change the inlet trace gas pressure and (or) concentration, a range of leak rate more than 6 orders of magnitude can be directly calibrated without extrapolation. Experimental result shows that Helium leak rate lower than 10^{-14}Pa*m^3/s has been detected and calibrated reliably by this system.

[1]Liangzhen Cha, Theoretical and experimental studies of a platinum wire-glass standard leak, Vacuum,Vol 41,PP:1860-1862(1990).

5:00pm **VT-WeA10 A Compact Leak Rate Calibration System for Both Pressure and Vacuum Modes**, ***Xu Chen***, *Ping Chen, Q. Zhang, Q. Liu, Liangzhen Cha*, Tsinghua University, P.R. China

To meet the quality control of the industrial leak detection, especially the growing demand in sniffing application, a compact leak rate calibration

system is developed to calibrate the leak rate from high pressure to atmosphere (pressure mode) and from atmosphere to vacuum (vacuum mode). A new method based on constant volume change in pressure for pressure leak rate calibration is utilized. A differential capacitance diaphragm gauge (CDG) is used to markedly decrease the temperature effect. It is found that $1*10^{-5}$ $Pa*m^3/s$ pressure leak rate at room temperature can be calibrated with an accuracy of better than ±5% (with confidential level 95%) and leak rate as low as $3*10^{-6}$ $Pa*m^3/s$ can be calibrated with an accuracy of better than ±15% with an environmental temperature change less than ±0.1°C. The total test period is about 30 minutes. This compact system is suitable for leak rate calibration in industrial environment for both pressure and vacuum modes.

Vacuum Technology
Room: C-104 - Session VT-ThM

Gas Dynamics and Flow
Moderator: R. Dobrozemsky, University of Vienna, Austria

8:20am **VT-ThM1 Cryopump Pumping Performance Estimation Using Flow Meter Method**, *H.-P. Cheng*, ***Y.-C. Lu***, National Taipei University of Technology, Taiwan, *M.-K. Hsu*, World Hold Engineering Consultants, LTD

The pumping performance of cryopump, ULVAC CRYO-U10PU, is estimated by the flow meter method, which is widely used in the estimation of the pumping capacity of the turbo molecular pump for inlet pressure greater than 1.0E-04 Pa. The test dome is designed according to the instruction of JVIS-005 and connected directly with the cyropump. The inner diameter of the test dome is the same as the inlet port of the pump. The PIRANI gauge (ULVAC GP-1000, range 0.4 to 2700 Pa) and the ION gauge (ULVAC GI-1000 WIT, range 1.3E-05 to 6.7E-01 Pa) are equipped on the peripheral of the test dome to measure the test dome pressure according to the instruction of the JVIS-005. Four flow meters (Sierra 820 series, ranges 25 SCCM, 500 SCCM, 10 SLM, and 50 SLM) hybrid the needle valves are used to control the inlet gas throughput of the pump and two types of gas, nitrogen and argon are introduced. The variations of the temperature of the first stage (80K) of the cryopump and the test dome pressure relative to the operating time of cryopump are discussed. The pumping speed and throughput of the cryopump versus to the test dome pressure that is considered as the inlet pressure of cyropump are plotted and compared to the values described in the catalog of ULVAC. Finally, the residual gas analysis is made to investigate the residual gas in the whole system. And the temperature recovery time is recorded after the cryopump is turned off. According to the measurements, the temperature of the first stage of cryopump is in the steady state condition after two hundred minutes of the starting of the cryopump. The pumping speed for nitrogen is near to the value shown in the catalog of ULVAC and the argon is nearly twice than the catalog. The main residual gases are hydrogen and water at the status of the ultimate pressure operating condition. The recovery time from normal operating temperature of the cryopump to the room temperature is over three hundred minutes after the pump turned off.

8:40am **VT-ThM2 Developing of Calculation Methods of Diffusion Vacuum Pumps' Characteristics**, *M.G. Sapeshinsky*, Bauman Moscow State Technical University, Russia, ***B.N. Kemenov***, NPK INTELVAC, Russia

Results on 3-dimensional mathematic simulations of processes in an inlet chamber of diffusion vacuum pumps, of interaction between pumped out gas and steam molecules are presented. Description of algorithmus and of a calculation programs complex for characteristics of diffusion vacuum pumps is given. Using the developed calculation programs the influence of parameters and form of a steam flow, geometry of a body, as well as of an oil reflector and protecting screens, on operation rate and on reverse oil flow of diffusion pumps is investigated. Rated and experimental data are compared. The concept of optimisation of an inlet chamber of diffusion pumps is developed

9:00am **VT-ThM3 Comparison between Monte Carlo and Analytical Calculation of the Conductance of Cylindrical and Conical Tubes**, ***J. Gómez-Goñi***, *P.J. Lobo*, Universidad Politécnica de Madrid, Spain

The accurate calculation of transmission probabilities of ducts in the molecular flow regime has become a need of vacuum standards on the ultra high vacuum region. The usual approach is to simulate a molecular flow of molecules with a Monte Carlo method, because of the difficulties that arise trying to find a solution of the Clausing integral equation in a system of a given geometry. As modern computers increase their speed, the accuracy of Monte Carlo methods is higher and higher. So it becomes important to compare simulation results with data obtained by other methods to check that random number generators used in the Monte Carlo are not biased. Moreover, with numerical methods we can obtain the density of molecules along the tube. This density is important in molecular beam formation studies. We have applied both a Monte Carlo method and numerical solutions of the Clausing integral equation for cylindrical and conical tubes. In the case of cylinders, we have found very accurate transmission probabilities solving numerically Clausing integral equations. The method consists in an approximation of the Clausing function with Chebyshev polynomials using a subroutine made by the Numerical Algorithm Group (NAG).[1] Comparing with other values found in the literature[2] made by a variational method, we have obtained values near the upper bound and quite far away from the lower bound. In the case of cones, we obtain values which agree with values found in the Literature[3] to a high degree of accuracy.

[1] NAG Subroutine D05AAF.
[2] R.J. Cole, J. Inst. Maths. Applics. 20, 107-115 (1977).
[3] R.P. Iczkowski, J.L Margrave and S.M. Robinson, J. Phys. Chem. 67,229 (1963)

9:20am **VT-ThM4 Modeling Molecular Drag Pumping in the 20th Century: A Personal View**, ***J.C. Helmer***, Consultant **INVITED**

In the last decade we have had a renaissance in molecular drag pumping, led by the design of hybrid turbopumps that exhaust at pressures above 10 Torr, to oil-free forepumps. The exhaust stages, which operate in laminar flow, may be of the Gaede, Holweck, or Siegbahn type. While Gaede provided the underlying model in 1913, upon which all are based, the Gaede design was not developed as a commercial pump until it was revived by Varian SpA, in Turin, in 1992. The neglect of the Gaede design was accompanied by 80 years neglect of the molecular drag model, in deficiencies which Gaede himself identified in his original papers. Pumps operate in continuum flow, which is a new field in vacuum science, extending the classical field of molecular flow. The appropriate mathematical tools have been developed in the fields of fluid mechanics (CFD), aerodynamics (RGD) and molecular simulation (DSMC). It is a challenge to extract from these fields a subset of theory that is appropriate and useful to molecular pumping. Many papers miss the fact that molecular drag pumps operate in the classic analytic regime of Couette-Poiseuille flow, and results need to be compared with the characteristics of this type of flow. Even G.A. Bird, author of the famous DSMC method, recommends that numerical simulations be guided by analytic models. Useful theory has been developed in the design of gas (journal) bearings, and floating magnetic recording heads. For molecular pumping, the inertial term in the viscous equations should not be neglected. Some papers calculate solutions with a form of slip-flow that has no physical correspondence, since in practice the molecules have full accomodation to the surface. This is an emerging field which has many opportunities for student research.

10:00am **VT-ThM6 Mathematic Simulation of Processes in Flow Parts of Hybrid Turbomolecular Vacuum Pumps**, ***M.G. Sapeshinsky***, *A.V. Ponomarev*, Bauman Moscow State Technical University, Russia

Results on 3-dimensional mathematic simulation of processes in flow parts of hybrid turbomolecular pumps (TMP) are presented. Description of algorithms and of a calculation programs complex for characteristics of hybrid turbomolecular pumps with molecular and drum forcing channels is given. The influence of geometric parameters of a flow part on an operation rate and a maximal compression degree of pump channels is investigated. The concept of optimisation of hybrid pumps under restrictions of controlled parameters is developed.The optimal variants of a flow part are given. Rated and experimental data are compared. There is developed an algorithm of optimization of a TMP flow part with axial and axial-radial gas flow under functional restrictions on controlled parameters, ensuring the desired TMP operation rate at fixed suction pressure for a chosen gas, and also the desired operation rate for several gases using the algorithm of slipping access and the absolute penalty functions metod. It is found out, that within the investigated operation rate's range an axial-radial scheme has better mass-size characteristics compared to that of an axial scheme. Decrease in volume of a flow part with optimal geometric parameters makes from 36 % (S=0.5 m^3/sec) to 53 % (S=20 m^3/sec) due to decreasing the axial rotors number. Here the external diameter of axial rotors increased from 35 % (S=0.5m^3/sec) to 5 % (S=20m^3/sec). More preferable is the axial-radial scheme with periphery-center flow direction. It is found out, that if ensuring the desired raised evacuation characteristic for light gases, the flow part volume of all the three schemes increases due to increase in axial rotors number and smoother changing of geometric parameters over rotors, and mass-size characterictics of axial-radial schemes come worse. Decrease in the flow part volume comparing to that of an axial scheme makes for the scheme with periphery-center flow 24 % (S=20m^3/sec, P=10^{-5} Pa, S=15 m^3/sec and P=10^{-3} Pa). The volume of an axial scheme raised on 93 %, of axial-radial schemes - on 163 % and 150 % compared to the computation results when ensuring the desired operation rate only for nitrogen.

10:20am **VT-ThM7 Two-Dimensional and Three-Dimensional Monte Carlo Simulation on the Pumping Performance of a Turbomolecular Pump with Rough-Surface Blades**, *M. Yabuki,* ***T. Sawada****, W. Sugiyama,* Akita University, Japan, *M. Watanabe*, Osaka Vacuum Ltd., Japan

The elements of a turbomolecular pump (TMP) are sometimes coated with ceramic (SiO_2) film for the purpose of preventing corrosion on the TMP. The blades coated with SiO_2 have relatively rough surfaces, and it has been confirmed by previous experiments that the SiO_2-coated TMP gives a higher maximum-compression ratio than the non-coated TMP. This paper describes the Monte Carlo simulation on the effects of the surface roughness of blades on the pumping performance of a TMP. The surface roughness was measured by SEM, and then the distribution of roughness slope angles was obtained from the surface roughness data. The surface roughness was modeled by statistically located circular-conic peaks and dimples of the same base radius and various base angles. The base angles were sampled in a statistical manner so as to accord with the measured slope angle distribution. Both the 2D and 3D Monte Carlo simulations were done in the free molecule flow regime and the simulated maximum-compression ratios were compared with the previously measured values. The 3D simulation naturally showed a better agreement with the measured values than the 2D simulation. However, it was proved that the 2D simulation gave reasonable results with much less computation time than the 3D simulation when the ratio of blade height to pitch was larger than the unity.

10:40am **VT-ThM8 Experimental Analysis of Tapered Gaede Pumps**, ***S. Giors****, R. Gotta, J.C. Helmer,* Varian S.p.A., Italy

In 1993 Varian S.p.A. revived the Gaede design and developed it for commercial hybrid turbomolecular pumps. The Gaede stages exhaust at pressures above 10 Torr, operating in laminar viscous flow. Classical Gaede stages are characterized by uniform cross section of the channel, from inlet to exhaust. Both modelling and experimental analysis agrees in showing that the maximum compression in the viscous regime is inversely proportional to the square of the height h between disk and channel surface, while channel pumping speed is proportional to the inlet cross section of the channel and hence to h. From this reasoning comes the simple idea that tapering the height h of the channel from inlet to exhaust, keeping the same entrance section, can greatly increase compression, without compromising the pumping speed of the channel. An experiment was designed to test these concepts. A uniform channel and one with a 3:1 taper, with the same inlet channel section, are compared in viscous conditions. Results show that the channels must be compared not just in terms of maximum compression and maximum speed, but in terms of the complete speed vs. compression characteristics. Actually the tapered channel shows a slight decrease in maximum pumping speed, but that is highly compensated by increased compression and improved shape of the speed vs. compression characteristics. Another promising advantage of the tapered channel with respect to the uniform one, is reduction in power dissipation with the same exhaust pressure and flow. Comparison of experimental results with model calculations, indicates some ways in which the Couette-Poiseuille model needs to be improved.

11:00am **VT-ThM9 Pumping Performance Investigation of a Turbo Booster Vacuum Pump Equipped with Spiral-Grooved Rotor and Inner Housing by CFD Method**, ***H.-P. Cheng****, C.-P. Chien,* National Taipei University of Technology, Taiwan, *C.-P. Lee*, China Engineering Consultants, INC

This paper estimates the pumping performance of a turbo booster vacuum pump equipped with spiral-grooved rotor and inner housing by CFD method. The computational domain hybrids the flow channels in the rotor and inner housing, thus the gas can flow continuously from the inlet of rotor through the outlet then into the inlet of inner housing and exist. The pumping characteristics for the spiral angle of the rotor, and the groove number and the spiral angle of the inner housing are detailed investigated. The spiral angle, 15 degrees, of rotor can adequately increase the effective pumping length of the flow channel and overcome the following increased friction force, therefore the pumping performance can be enhanced. Similarly, the spiral-grooved inner housing with 5 grooves and spiral angle 27 degrees can also have the better pumping performance. The results also indicate that there is an axial vortex dominated the spiral-grooved flow channel of both rotor and inner housing. The back stream from rotor channel to the vacuum chamber is evident. Otherwise, the detailed three-dimensional velocity vector, axial velocity contour, and pressure fields are shown in the paper. The arithmetic axial pressure distribution, compression ratio are also discussed. The pumping performance of two prototypes are also measured and shown in the paper. The discrepancies among experiments, simulation, and expected target are discussed.

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