Vacuum Technology Division
Room: 7 & 8 - Session VT+MN-MoM

Progress with Measurement in Vacuum

Moderators: Martin Wüest, INFICON Ltd., Liechtenstein, Steve Borichevsky, Applied Materials, Varian Semiconductor Equipment

8:20am **VT+MN-MoM1 New Vacuum Standard by Ultra-Precise Refractive Index Measurement**, ***Jay Hendricks***, *J.E. Ricker, J.A. Stone, P. Egan, G.E. Scace, K.O. Douglass, D.A. Olson, G.F. Strouse*, NIST

NIST has now completed the 5th year of an Innovations in Measurement Science (IMS) initiative with the aim of developing a new paradigm in the methodology of pressure and vacuum measurement and primary standards. The research program has now successfully developed a new standard that is based on the ultra-precise measurement of gas refractive index. This advance now enables NIST to replace mercury manometer standards with a new quantum-based, photonic technique. The new standard, is based on the fundamental physics of light interacting with a gas, and when the gas is helium, the refractive index is based upon first principle quantum chemistry calculations and is realized as a primary standard. For the vacuum community, a photonic realization of the pascal represents a fundamental change in how the unit of pressure is realized in that it will be directly related to the density of a gas by the temperature, refractive index, and Boltzmann constant. The photonic technique has now achieved important benchmarks in performance when compared to the existing primary standards based on mercury manometers: The photonic technique has a 20x smaller footprint, 100x faster sensing response time, extended to 100x lower pressure, a tenth of a mPa resolution over the full range, and has demonstrated impressive accuracy, reproducibility and hysteresis for an emerging technique [1]. Data will be presented that shows this technique has now reached or surpassed mercury manometer performance which creates a new paradigm for vacuum metrology and realization of the SI unit, the pascal. Future NIST work will explore improvements that will enable the device to become a portable pressure and vacuum standard for international key comparisons in pressure and vacuum metrology.

[1] Comparison measurements of low-pressure between a laser refractometer and ultrasonic manometer, Review of Scientific Instruments, Volume 87, May 2016

Accepted: May 2016, Issue 5, 10.1063/1.4949504

8:40am **VT+MN-MoM2 Construction and Testing of the NIST Variable Length Optical Cavity Pressure Standard**, ***Jacob Ricker***, *J. Hendricks, G.E. Scace, P. Egan, J.A. Stone*, NIST

NIST is constructing and testing a new refractometer, referred to as the Variable length optical cavity (VLOC), that will redefine how pressure and vacuum is measured. NIST has shown in previous talks and papers that this technique will replace all mercury manometers in the near future. However, the critical final piece of this project is to base the traceability of pressure measurements to fundamental constants of the universe and not on the physical artifacts like mercury density in a manometer. Theoretical quantum mechanics have been used to precisely calculate the refractivity (n-1) of a gas. NIST will experimentally verify these calculations and provide experimental measurements of refractivity for other gasses/mixtures.

The engineering of the VLOC will be discussed along with limitations and technical complications that have arisen. Specifically, the distortions of the optical cavities and methods to overcome these limitations. Additionally, the steps required to maintain ultra-high purity gas will be discussed. Finally, the testing and final steps to achieve full operation will be discussed and the relation to the 2018 redefinition of the Boltzmann constant.

9:00am **VT+MN-MoM3 Fast-Switching Dual Fabry-Perot Cavity-based Optical Refractometry – A Powerful Technique for Drift-Free Assessment of Gas Refractivity and Density**, ***Ove Axner***, *I. Silander, T. Hausmaninger*, Umeå University, Sweden, *M. Zelan*, RISE Research Institutes of Sweden, Sweden **INVITED**

Since pressure has a temperature dependence it is not trivial to accurately assess gas amounts by pressuring measuring devices. However, the (number) density does not suffer from such limitations. Optical Refractometry (OR) is a powerful technique for assessment of gas refractivity and density. The highest resolution is obtained when performed in a Fabry-Perot (FP) cavity. In FP-Cavity based OR (FPC-OR) the change in the frequency of laser light, locked to a longitudinal mode of a FP cavity, is monitored while the amount of gas in the cavity is being changed. Since frequency is an entity that can be assessed with enormous precision, the precision of FPC-OR can be extremely high. However, although potentially very powerful, FPC-OR is often limited by thermal deformation of the spacer between the mirrors. A partial remedy to this is to use two FP cavities, termed Dual FPC-OR (DFPC-OR).

We have prophesied that if measurements could be done under drift free conditions, the technique would be able to circumvent most of these limitations. A possible strategy for drift-free DFPC-OR, termed fast switching DFPC-OR (FS-DFPC-OR), is presented in which measurements are made under such short times that the drifts of the cavity can be disregarded. The methodologies developed circumvent the problem with volumetric expansion, i.e. that the gas density decreases when gas is let into the measurement cavity by performing a pair of measurements in rapid succession; the first one assesses the density of the gas transferred into the measurement cavity by the gas equilibration process, while the second automatically calibrates the system with respect to the ratio of the volumes of the measurement cavity and the external compartment. The methodologies for assessments of leak rates comprise triple cavity evacuation assessments, comprising two measurements performed in rapid succession, supplemented by a third measurement a given time thereafter.

We predict that refractivity and density can be assessed, under STP conditions, with a precision in the 10^{-9} range. The absolute accuracy is expected to be given by the calibration source. If characterized with respect to an internal standard, the accuracy can be several orders of magnitude better. The temperature dependence of FS-DFCB-OR is exceptionally small, typically in the 10^{-8} - 10^{-7}/°C range, primarily caused by thermal expansion of the spacer material.

A first realization of a FS-DFCB-OR set up for assessments of gas refractivity and density will be presented and its performance will be demonstrated. We will discuss how to design an FS-DFCB-OR system for optimal performance for assessments of gas refractivity and density.

9:40am **VT+MN-MoM5 Cold Cathode Gauge Improvements Extend Performance into UHV Pressure Range**, ***Timothy Swinney***, *G. Brucker*, MKS Instruments, Inc., Pressure and Vacuum Measurement Group

Cold cathode gauges (CCG) of inverted magnetron design are routinely used to measure pressure in industrial high vacuum chambers. Reduced internal outgassing, compared to hot cathode gauges, also makes CCGs well suited for accurate ultra-high vacuum (UHV) measurement in applications such as high-energy physics, surface science experiments and ultrahigh resolution mass spectrometers. In order to provide accurate and repeatable pressure measurements extending into deeper UHV levels, it is important to design CCGs that provide a consistent linear response to pressure over the entire measurement range. Our latest research efforts have focused on the understanding of gauge signal response to pressure with particular emphasis on the displacement of the magnetron knee and discharge sustain issues to lower pressures through systematic design changes. In this presentation, the linear response of CCGs to pressure is explained based on a simple pure electron plasma model. Pressure readings below the magnetron knee are described in terms of a pressure-dependent plasma model controlled by design parameters. The effect of magnetic strength, electric field and plasma boundary conditions on the onset of the magnetron knee and the ability to sustain a stable discharge into UHV levels is described. A patent-pending modification to the CCG internal electrode design is presented that extends the operational pressure of the gauge into deeper UHV levels by controlling the location of the pure electron plasma inside the ionization volume. This new understanding of CCG signal response to pressure has led to the development of enhanced sensor designs that operate at pressures one to two decades lower than legacy designs.

10:00am **VT+MN-MoM6 Sapphire MEMS based Capacitance Diaphragm Vacuum Gauge for 0-0.1Torr Operating at 200 °C**, ***Takuya Ishihara***, Azbil Corporation, Japan, *M. Sekine, M. Soeda, M. Nagata*, Azbil Corporation

To meet with downsizing of semiconductor device, various new manufacturing processes such as Atomic Layer Deposition (ALD) and Atomic Layer Etching (ALE), are put into practical use. In particular, ALE is a new atomic level etching technique, which can be applied to high aspect ratio structure or narrow slit . Conventionally, the pressure range of capacitance manometer for etching process is mainly 0-0.1Torr for reasons such as using inductively coupled plasma. And self-heating temperature of that is usually 45 °C, or at most 100 °C for the stabilization.

In this paper, authors have assumed that towards ALE process enhancement, etching would require high temperature process operation, such like 200 °C to prevent by-product from depositing inside of manometer in deposition step. Therefore, there is a motivation to develop capacitance manometer with its pressure range of 0-0.1Torr operating at 150-200 °C.

Entirely sapphire-based capacitive pressure sensor chips utilizing MEMS (Micro-Electro-Mechanical Systems) processes, which can be operated at 200 °C with from 0-1 to 0-1000 Torr pressure range have been developed by authors (Fig.1). To diminish pressure range to 0-0.1Torr, we need to reduce sensor diaphragm thickness to get sufficient sensitivity, but thinner diaphragm would be influenced heavily by noises, such like vibration from vacuum pumps, diaphragm sticking, and mechanical stress from sensor package and so on.

One of the critical issue is the zero point drift which was observed under back ground vacuum level after applied pressure over 100 °C (Graph.1). As a result of various verification experiments, this phenomenon was proved to be caused by the slight difference of temperature between sensor diaphragm surface and dilute gas in back ground vacuum. In other words, thermal energy exchange between diaphragm and gas results local expansion or shrinkage of the diaphragm because of its thinner thickness, which deform diaphragm (Fig.2). In our thermal simulation like Fig.3, only 0.05 °C temperature deference causes 0.5% Full Scale zero point drift at 0.1Torr range, which is fatal for the monitoring or controlling of the process. The temperature of the background gas depends on the temperature of inside wall of the process chamber which cannot be controlled by capacitance manometer itself. To solve this problem we have developed new sensor chip structure utilizing sapphire MEMS technology in which the process gas exchanges thermal energy with sensor chip before arriving to the diaphragm (Fig.4). By this new sensor chip, the zero point drift was suppressed to under 0.1% Full Scale, which is sufficient value to apply for the processes (Graph.1).

10:40am **VT+MN-MoM8 ROSINA/Rosetta: Exploring the Origin of our Solar System with Mass Spectrometry in Space**, ***Kathrin Altwegg***, University of Bern, Switzerland **INVITED**

On 30 September 2016 the European Space Agency's Rosetta spacecraft softly crash-landed on comet 67P/Churyumov-Gerasimenko and brought an intense period of more than 2 years of continuous investigation to an end. Rosetta data led to many discoveries about the origin of the material and the processing in our early Solar System. Among the payload instruments, ROSINA, the mass spectrometer suite, obtained fundamental properties of the comet by measuring the gases emanating from its nucleus.

Besides detecting many organic molecules never seen in space before, ROSINA was also able to measure precise isotopic abundances for noble gases, sulfur and silicon as well as D/H in water and H2S. By following the comet from 3.8 AU to perihelion and out again to 3.8 AU desorption patterns could be followed for individual species, allowing deeper insights into the nature of cometary ice. Some of the findings clearly point to unprocessed ice from the prestellar stage which allows to study chemistry in the presolar cloud more or less "in situ".

Some of the most important findings will be discussed in the presentation like the "zoo" of volatile and semi-volatile organics, the isotopic signature of Xenon and its relation with the terrestrial atmosphere.

11:20am **VT+MN-MoM10 Stabilization of Emission Current from Cold Field Emitters by Reducing Pressure to 10^{-10} Pa**, ***Keigo Kasuya***, *T. Ohshima, S. Katagiri, T. Kawasaki*, Hitachi, Ltd., Japan

In the presence of a strong electric field, electrons are emitted from sharply pointed cathodes at room temperature. This cold field emission (CFE) process provides a prominent electron beam with high brightness and a low energy spread, so CFE emitters are used extensively in a variety of electron microscopes.

One of the important challenges for CFE is stabilizing the emission current. The adsorption of residual gases on the emitter increases the surface work function and decreases the emission current over time. Additionally, surface sputtering by ions causes irregular current fluctuations.

One way to stabilize emission current is to reduce the pressure around the electron gun. This decreases incident gases and ions hitting the emitter and slows the current decrease. We reduced the pressure of an electron gun from 10^{-8} to 10^{-10} Pa by using non-evaporative getter (NEG) pumps [1]. This stabilized the emission current so that it was almost constant over a 24 hour period. The 90% decrease time, the time it takes the current to fall to 90% of the initial value, was increased from 10 minutes to 1280 minutes. In addition, the maximum emission current was increased from 30 μA to 1000 μA. With this gun, operators can use electron microscopes without the need for emission current adjustment.

By applying this technology, we developed a 1.2 MV high voltage transmission electron microscope [2]. The electron gun is equipped with a preaccelerator magnetic lens for enhancing the effective brightness of the electron beam. The pressure of the gun was $3X10^{-10}$ Pa, and a stable emission current was obtained. The microscope achieved the world's highest spatial resolution of 43 pm.

Part of this research was funded by a grant from the Japan Society for the Promotion of Science (JSPS) through the "Funding Program for World-Leading Innovative R&D on Science and Technology (FIRST Program)," initiated by the Council for Science and Technology Policy (CSTP).

[1] K. Kasuya et al., J. Vac. Sci. Technol. B, 34, 042202 (2016).

[2] K. Kasuya et al., J. Vac. Sci. Technol. B, 32, 031802 (2014).

11:40am **VT+MN-MoM11 Measurement and Prediction of Quadrupole Mass Spectrometer Sensitivities**, ***Robert Ellefson***, REVac Consulting

Accurate analysis of partial pressure and gas composition by quadrupole mass spectrometry (QMS) requires measuring the QMS sensitivities and fragmentation factors for gas species of interest. The sensitivity is the ratio of ion current for the species to the partial pressure of that species. Fragmentation factors are ratios of fragment [#] ions to the parent ion and are used to correct for species interference at the fragment mass. Measurement with pure gases of each species is the traditional method for determining sensitivities and fragmentation factors; this involves a significant investment in gases and delivery hardware. The QMS ion source operates in the molecular flow regime so that for each species, gas flow is independent of other components present. This allows known mixtures of species to be used for independently measuring multiple species sensitivities. Data using two designed mixtures are presented giving sensitivities and fragmentation ratios for nine species: H_2, He, H_2O, N_2, O_2, Ar, CO_2, Kr and Xe. Gases are delivered to the QMS with a molecular flow inlet system which delivers a broad range of predictable partial pressures for species. Sensitivity versus partial pressure determines the range of linear operation of the QMS indicated by constant sensitivity for that species over a range of pressure. A model for predicting QMS sensitivities for species not measured is also presented. The model uses the QMS sensitivities for the known gas species analyzed to determine the parameters for predicting the sensitivity of an unknown. The factors of the model are: 1. A calculated [#] ionization cross section as a function of incident electron energy data from the NIST Web Book, 2. The mass spectrum of the gas from the Web Book, 3. A model for ion transmission as a function of mass for the QMS in use, and, 4. The QMS sensitivity for N_2 as a reference point to capture the ion source geometry and unique behavior of the QMS under test. Examples of the predictive method and estimated uncertainty are given.

Vacuum Technology Division
Room: 7 & 8 - Session VT-MoA

Material Outgassing, Adsorption/Desorption and XHV

Moderators: Giulia Lanza, SLAC National Accelerator Laboratory, Jacob Ricker, NIST

1:40pm **VT-MoA1 Weight of Water on the Solid Surface in Air and Vacuum**, ***Richard Green***, National Research Council of Canada, Canada **INVITED**

In 2018, after over 30 years of research by national measurement laboratories around the globe, the unit Kilogram is expected to be redefined in terms of a fundamental constant of nature; Planck's constant. The present definition has not significantly changed in over 120 years and relates to a single cylinder made of exactly one kilogram of platinum-iridium alloy that is stored in a vault in Sèvres, France. In order to connect the present Kilogram measured in air to a redefined Kilogram measured in vacuum, new tools and methodologies have been developed to understand and quantitatively determine the change in mass that occurs when metals are placed in vacuum. While initially concerned with platinum, work has extended to stainless steel and other surfaces. With resolutions on the order of a 100 parts per trillion possible, sorption of less than 0.01 monolayers of water is observable.

In this talk we will present measurement techniques and tools used to quantitatively and traceably determine the weight of water and hydrocarbons desorbed from a surface as it is exposed to vacuum. The techniques have been used to study factors such as pressure, surface roughness and contamination, which influence the quantity and dynamics of desorbed mass. The investigations will be presented in context of efforts at the National Research Council of Canada to make the world's most accurate measurement of Planck's constant.

2:20pm **VT-MoA3 Hydrogen Measurement using a Thermal Desorption Spectrometer**, ***JongYeon Lim***, Korea Research Institute of Standards and Science, Republic of Korea, *K.D. Kim, H.S. Oh, C.H. Lim, Y.D. Joh*, Infinity Vacuum Technology, Republic of Korea

The TDS system, designed for measuring outgassing rate of a diameter of 10 mm sample, with two pumping paths; one is ordinary throughput path with an orifice, the other a UHV path, has been newly developed to measure any molecules on samples with a sophisticated DAQ system.

Two paths are directly connected to the main chamber equipped with devices including a rod-guided halogen heater allowing the sample temperature up to 1200 ºC .

The throughput path utilizes the UHV equipment to measure the outgassing rate quantitatively and qualitatively. In the case of quantitative hydrogen measurement, the throughput path does not have enough pumping speed since a small orifice diameter has the conductance limitation.

The experimentally acquired system calibration factor for the throughput method is 16.0, which is defined as the ratio of background outgassing rates with the gate valve closed and open using the throughput method.

In order to verify the measurement reliability a NIST Standard Reference Materials (SRM) was introduced to the TDS system. Most of hydrogen was desorbed during the course of heating process up to 800 ºC for 90 minutes. The area under the H2 peak is proportional to the nominal value of 126.8 wt ppm (uncertainty of 2 %). The hydrogen calibration factor of 4.38686E23 was realized. The system claims the hydrogen measurement resolution of 1.3E-6 wt ppm.

In this presentation we briefly introduce the accurate UHV outgassing measurement system for both qualitative and quantitative analyses, which has an 18 % uncertainty of total outgassing rate with Inficon BPG400 HV gauges and a unique self-calibration function.

Acknowledgements: Results are partially attributed to two national projects sponsored by the Korean Ministry of Trade, Industry & Energy, and the KRISS main project (Contract Nos. 10048806, and 170111649).

2:40pm **VT-MoA4 Automatic Flowmeter and Dynamic Expansion System for UHV/XHV Studies**, ***James Fedchak***, *J. Scherschligt, D. Barker, S. Eckel*, NIST

NIST is presently creating the Cold Atom Vacuum Standard (CAVS), a quantum-based fundamental primary vacuum sensor which significantly departs from present methods of measuring and realizing ultra-high vacuum (UHV) and extreme-high vacuum (XHV). The CAVS is an absolute sensor based on the loss-rate of ultra-cold atoms from a conservative magnetic trap due to collisions of the trapped cold atoms with the ambient background gas, and will cover a pressure range of 10^{-8} torr to below 10^{-12} torr, thus spanning UHV and into XHV. Knowledge of the thermalized collision cross-section, or loss-rate coefficient, between the trapped ultra-cold sensor atoms and the background gas is critical to operation of the CAVS; such collisions are also important in other experiments and devices based on trapped cold atoms because they limit the lifetime of atoms in the trap and, in addition, glancing collisions can increase statistical and other uncertainties. To support the CAVS and other atomic physics programs, NIST is developing a dynamic expansion system to set a known pressure in the CAVS, which will allow the experimental determination of collision cross-sections, relative gas-sensitivity factors, and facilitate studies of other systematic effects. To that end, we are developing a constant pressure flowmeter capable of producing flows of at least 10^{-13} mol/s and a dynamic expansion system to produce pressure rises as small as 10^{-10} Pa. The system is designed to produce low gas-flows of H_2 and many other gases of interest, to be fully automated, and to have extremely low outgassing rates. The design and construction of the UHV/XHV flowmeter and dynamic expansion system will be discussed.

3:00pm **VT-MoA5 Development of a New UHV/XHV Pressure Standard (Cold Atom Vacuum Standard)**, ***Julia Scherschligt***, *J.A. Fedchak, S. Eckel, D. Barker*, NIST **INVITED**

NIST has a long history of laser cooling and trapping of neutral atoms, largely motivated by building better time standards or clocks, and has recently begun a program to extend the metrological capabilities of cold trapped atoms to measurement of vacuum. This will align vacuum metrology to the emergent NIST "Quantum SI" paradigm, in which a measurement has intrinsic traceability and the line between sensor and standard is blurred. Since the earliest days of neutral atom trapping it has been known that the background gas in the vacuum limits the lifetime of atoms in the trap. We are inverting this problem to create a quantum-based standard and sensor. Indeed, because the measured loss-rate of ultra-cold atoms from the trap depends on a fundamental atomic property (the loss-rate coefficient or thermalized cross section) such atoms can be used as an *absolute* sensor and *primary* vacuum standard. Researchers have often observed that the relationship between the trap lifetime and background gas can be an indication of the vacuum level, but a true absolute sensor of vacuum has not yet been realized. This is because there are many technical challenges that must be overcome to create a device that's truly absolute and primary. The NIST program addresses these challenges both theoretically and experimentally: we have begun *ab initio* calculations of collision cross sections between the trapped cold atoms and the background gas and, on the experimental side, we are thoroughly investigating the systematic uncertainties associated with using an atom trap to determine vacuum level, particularly those associated with loss mechanisms (in a non-ideal trap) other than due to background collisions. We are designing and building the apparatus to measure relevant cross sections, and building our first prototype vacuum sensing apparatus. In this presentation, we will discuss our theory progress, and present our newest measurements, as well as discuss how the Cold Atom Vacuum Standard fits into the broader picture of the NIST dissemination of the Quantum SI.

4:00pm **VT-MoA8 VTD Early Career Award Invited Talk: Modern Metrology Practice for Calibration and Reliability Testing of Vacuum Measurement Products**, ***Scott Heinbuch****, MKS Instruments, Inc. **INVITED**

Vacuum technology is traditionally very slow moving in terms of new techniques and innovations. The measurement techniques of today don't differ drastically from 20 years ago or even longer. That does not mean there aren't new ways we can think about how we use that measurement technology in a metrology lab today. Often times, critical vacuum measurements take time; time being a taboo word in today's product development discussions. There are many practices to help us reduce time waste while still ensuring good vacuum measurement. Principles of Lean have helped us to reduce types of waste in our lab creating an efficient environment where experiments can be set up accurately and timely. Similarly, Six Sigma ideas have given us a set of tools and a common language to quickly identify root cause for problems and simplify our data analysis techniques. These same tools have helped us to identify critical parameters for measurement systems analysis and better understanding of our measurement uncertainties when making measurements compared to our best in class Spinning Rotor, Stabil-Ion®, and Baratron® reference transducers. To further simplify our lab experience, vacuum system complexity has been reduced by an effort to separate the vacuum control system from our test

*** VTD Early Career Award**

development in which any user can interface with a vacuum system in the same way they would interface with a common laboratory instrument whether manually, or through a programming interface. Finally, none of this matters unless we are making our measurements with our customers in mind. Several customer applications have been reproduced and turned into standard tests for our products to improve our customers experience and our overall product performance and reliability.

4:40pm **VT-MoA10 Outgassing Rate Measurements of New Materials at NIST**, ***Makfir Sefa***, *J.A. Fedchak, J. Scherschligt, A. Zeeshan*, NIST

The Thermodynamic Metrology Group at NIST is interested in investigating the outgassing rates and gas uptake properties of a variety of materials. The motivation of these investigations is to support programs aimed to develop new vacuum, pressure, and temperature sensors. For example, materials with ultra-low outgassing rates are necessary to develop a cold-atom vacuum standard (CAVS), which is a metrological-quality XHV/UHV sensor based on ultra-cold atom technology. Chambers and vacuum components used in the CAVS must have ultra-low outgassing rates to achieve UHV and XHV backgrounds. The group is also interested in developing embedded temperature and pressure sensors, where the outgassing or gas uptake properties could influence sensor performance or applicability. In addition, the gas uptake properties of unique materials can be exploited for gas sensors or storage. This talk will highlight the outgassing measurements of heat-treated stainless steel chambers and 3-D printed stainless steel and titanium chambers. In addition, absorption and desorption of gases in 3-D printed nano-composite plastics will be discussed.

5:00pm **VT-MoA11 Scaling up an Ion Implant Process Chamber Cryopumping for 450mm Wafer Processing**, ***Steve Borichevsky***, Applied Materials, Varian Semiconductor Equipment

The semiconductor industry recently explored scaling up the wafer diameter from 300mm to 450mm. Ion Implant faced the challenge of providing process vacuum conditions for larger dopant ion beam currents and coping with the outgassing cause by the ion beam striking increased area covered by photoresist. The process chamber, where the ion beam strikes the target wafer, posed the most difficult vacuum challenges. The increased wafer size was predicted to generate 2.25 times the normal gas loads which would require nine 320mm cryopumps. In order to meet the requirements of implant, three prototype 500 mm diameter cryopumps were mounted onto a process chamber and tested. This presentation describes the basic architecture of an ion implanter, the decisions that lead up to the use of 500mm cryopumps, the results of the initial vacuum system testing and the Monte Carlo simulations.

Vacuum Technology Division
Room: 7 & 8 - Session VT-TuM

Large Vacuum Systems

Moderators: Jason Carter, Argonne National Laboratory, Gerardo Brucker, MKS Instruments, Inc., Pressure and Vacuum Measurement Group

8:00am **VT-TuM1 The Role of Vacuum Technology in Discovering the Gravitational Waves from Merging Black Holes**, *R.F.M. Weiss, **Michael Zucker***, LIGO Project Caltech and MIT **INVITED**

The observation of the gravitational waves from the merger of two black holes involved measuring the motion of mirrors to a precision of 10^{-18} meters over a distance of 4 km. The measurement was made by optical interferometry between suspended mirrors. Vacuum of 10^{-9} torr was required in 1.2 meter diameter 4km long beam tubes to avoid phase noise of the light and equivalently high vacuum was needed to avoid thermal noise of the mirrors from molecular collisions. The residual hydrocarbon background had to be controlled to avoid contamination of the optics. A significant challenge was to design and construct the vacuum system economically. Some of the fundamental physics and the engineering of the system will be described.

8:40am **VT-TuM3 Vacuum System Engineering for Cornell Brookhaven ERL Test Accelerator**, ***Yulin Li***, *D.C. Burke, B. Johnson*, Cornell Laboratory for Accelerator-Based Sciences and Education

A novel electron accelerator, Cornell Brookhaven ERL Test Accelerator (CBETA), is under development by a collaboration between Cornell Laoratory of Accelerator-based Sciences and Education (CLASSE) and Brookhaven National Laboratory. As a prototype accelerator for eRHIC, many unique accelerator technologies will be tested in CBETA, including photo-cathode electron injector, 4-turn superconducting RF (SRF) Energy Recover LINAC (ERL), non-scaling Fixed-Field Alternating Gradient (NS-FFAG) optics with 4x energy acceptance. The CBETA layout consists of an existing photo-cathode injector with SRF cryomodule (ICM) and a main LINAC cryomodule (MLC), a NS-FFAG return loop that transports electron beams at four energies, 42, 78, 114 and 150 MeV in single bore beampipe, and two splitter sections where the four energy beams are separated. The total circumference of CBETA loop is about 80-m. The basic requirement of CBETA vacuum system is to achieve adequate level of vacuum and physical aperture for transporting electron beams at four different energies. Furthermore, by the nature of test accelerator, the vacuum system engineering must accommodate a very high density of beam diagnostics tools, such as 100+ beam position monitors, beam profile viewers, etc. Beam path length up to 20° RF-phase is required in the splitter sections. Aluminum alloy is chosen for beampipe construction material for its good electric conductivity (resistive-wall), no residual radioactivity (from beam losses), low magnetization (from cold work and welding etc.) as well as lower cost of fabrication (machining, extrusion, etc.). Compact non-evaporable getter (NEG) pumps are used due to the space constraints. As in situ beampipe bakeout is practically impossible, a program was carried out to measure aluminum alloy outgassing rates at various controlled processing (bakeout, purified dry nitrogen venting, etc.). As a measure of vacuum system cost reduction, metal knife-edge seal flanges made of non-coated aluminum alloy (type 6013-T6) were developed as the beam diagnostics ports. The results of the outgassing study were used to validate vacuum pumping system design via 3D simulation. In this presentation, we report the status of CBETA vacuum system design and fabrication. Measurements of aluminum alloy outgassing rate and tests of aluminum knife-edge flanges will also be discussed.

9:00am **VT-TuM4 Vacuum System for CHESS-U Upgrade at CESR**, ***Xianghong Liu***, *S. Barret, D.C. Burke, J.V. Conway, A.T. Holic, Y. Li, A. Lyndaker*, Cornell Laboratory for Accelerator-Based Sciences and Education

A sextant of Cornell Electron Storage Ring (CESR) will be upgraded with Double Bend Achromat (DBA) lattice and CHESS Compact Undulators (CCUs) to significantly boost the performance of Cornell High Energy Synchrotron Source (CHESS). A lot of efforts are being made in preparation for the final installation in late 2018 for this upgrade project, dubbed CHESS-U. With this upgrade, CESR will be converted from a counter-propagating two-beam ring to a single-beam ring. The beam energy will be increased from 5.3 GeV to 6 GeV, and the beam current for normal operation will be increased from 120 mA to 200 mA. Because of the geometrical constraints from the magnets, the beampipe aperture of this new section is 52mm (horizontal) by 22mm (vertical), which is much smaller than the rest of CESR. The vacuum pumping for this new section will be different too, consisting of a combination of Non-Evaporable Getter (NEG) strips, modular NEG pumps, and ion pumps.

The beam pipes are mainly made of three types of aluminum extrusions, fitting inside quadrupole magnets, dipole magnets, and undulators respectively. All extrusions have cooling water channels to handle the thermal load from synchrotron radiation. The same channels are also used for vacuum hot-water bake-out. Dipole extrusions are formed to the correct bending radius by stretch forming in a softer temper, and heat treated to higher temper after forming. With required space available, the dipole extrusion includes an ante-chamber for NEG strip (SAES St 707) pumping. The NEG strip is activated by resistive heating to about 400°C for half an hour. The electrically insulating support mechanism of the NEG strip is adapted from the design used in APS. Instead of being built from extrusions, the dipole chamber where the X-ray beam exits is made of two machined halves that are welded together; a cylindrical crotch is inserted at the flared end of this chamber to absorb approximately 4 kW of synchrotron radiation.

In this presentation, we will give an overview of the design of the vacuum system, and report the estimated pressure profile based on Molflow$^+$ calculations, some design details of major components, results from NEG strip pumping tests, and progress in vacuum chamber productions.

9:20am **VT-TuM5 Newly Designed Alumina Ceramics Beam Pipe with Large Aperture for RCS in J-PARC**, ***Junichiro Kamiya***, *M. Kinsho*, Japan Atomic Energy Agency, *K. Abe*, HIPSD, Japan

The 3 GeV Rapid Cycling Synchrotron (RCS) in J-PARC aims to generate one of the highest power protons in the world. The design extraction beam power is 1 MW, which consists of 8.3×10^{13} protons per bunch with 3 GeV energy at 25 Hz repetition rate. The rapid change of the magnetic field at such repetition rate causes the induced current if the beam pipe was made of metal. Therefore, beam pipes of alumina ceramics were used. The cross-sectional diameters of the pipes range from 250 to 500 mm, as is the case for the titanium beam pipes and bellows. There are several cross-sectional shapes corresponding to the various beam shapes. The beam pipes in the dipole and quadrupole/sextupole magnets have racetrack and circular shapes, respectively. The beam pipes in injection magnets have racetrack and rectangular shapes. Unique cross-sectional shape is adopted for the ceramics beam pipes for the injection quadrupole magnet. Because the injection beam and circulating beam pass through the injection quadrupole magnet, its cross-section has a racket shape fitting into 500 mm diameter. Recently this ceramics pipes for the injection quadrupole magnets were newly designed. In the new design, the bellows are attached to the titanium sleeve of the beam pipe by welding to obtain the better maintainability in the narrow area under high radioactivation level. In the conference, we will report the design concept of the new alumina ceramics beam pipes with unique shape and the several results of the verification tests.

9:40am **VT-TuM6 Vacuum Performance of Taiwan Photon Source Storage Ring**, ***Hsin-Pai Hsueh***, *G.Y. Hsiung, J.R. Chen*, National Synchrotron Radiation Research Center, Taiwan, Republic of China

The Taiwan Photon Source storage ring vacuum system has been developed to be pre-baked and installed under vacuum for 14-m arc sections. The straight sections were in-situ baked followed the installation of adjacent arc sections. During first stage commissioning, foreign object was found and the bending vacuum chamber of this particular arc section was replaced without baking (neither pre-baked and installed under vacuum, nor in-situ baking). Subsequently, four more bending chambers in other four different arc sections were replaced without baking either. Since this time saving method is different from our system design philosophy, a detailed pressure and mass spectrum analysis is necessary. From measured data, the photon stimulated desorption is no longer as dominant as we would like it to be as designed. The thermal outgassing is more than 25% of total outgassing at highest current (400mA or above). The total beam dose is almost 1500 Amp-hour. The photon-stimulated-desorption (PSD) pressure over beam current ($\Delta P/I$) is 2.33E-11 Pa/mA. In this presentation, the analysis results will be presented.

11:00am **VT-TuM10 The Vacuum System Design of a New FEL Test Facility (CLARA) at STFC Daresbury Laboratory**, ***Keith Middleman***, STFC, UK

Recent UK government funding has facilitated the construction of Phase 1 of a unique FEL accelerator test facility (CLARA – Compact Linear Accelerator for Reserach Applications). This test facility will allow the UK to reserach a variety of FEL operating modes to establish a roadmap for the UK and its plan to build a UK FEL user facility.

This paper will look specifically at the vacuum system design of this unique accelerator detailing the challenging design of the photoinjector, the FEL section and the possible use of NEG coating and the requirement for

implementing a differential pumping scheme to separate two vacuum systems with 3 orders of magnitude pressure difference over 50 cm. Many other aspects of the accelerator design will be described and data showing the 'real' perfromance of the accelerator presented.

11:20am **VT-TuM11 EBL2: Realization and Qualification of an EUV Exposure System**, ***Michel van Putten***, *N.B. Koster, A.F. Deutz, B.A.H. Nijland, P.J. Kerkhof, P.M. Muilwijk, B.W. Oostdijck, J. Westerhout, C.L. Hollemans, E. te Sligte, W.F.W. Mulckhuyse, F.T. Molkenboer, A.M. Hoogstrate, P. van der Walle, J.R.H. Diesveld, A. Abutan*, TNO, Netherlands

In 2014, TNO started the design of a new facility to test samples with EUV light (13.5 nm). This facility is called EUV Beam Line 2 (EBL2) and enables study of the interaction of lithographic photomasks, optics and other samples with EUV light by irradiation, *real-time* imaging ellipsometry and *in vacuo* XPS surface analysis.

The integration of EBL2 started in Q2 of 2016 and at the end of 2016 the major milestone – *first light* – was reached. Currently, the building phase of EBL2 being completed. During Q3 of 2017 we anticipate to complete the qualification. Once qualified, the EBL2 facility will be accessible for external customers and research groups.

The realization of such a facility is the translation from a design to a manufactured, integrated and tested system. During AVS63 the realization of some of the modules was presented, this presentation will discuss the integration of the complete system. Challenges in this are: motion in vacuum, use of UHV compatible materials, alignment of EUV optics.

The qualification of the EBL2 facility was done in three steps: module verification, system verification, and system validation. During the verification steps, EBL2 and its constituent modules were assessed against specifications. Next was system validation where the ability to satisfy user needs was verified.

This presentation will focus on the completion of the realization of EBL2 and results of the system verification and validation phases.

EBL2's EUV irradiation performance is discussed in terms of EUV power, vacuum cleanliness, positioning of samples like reticles, and the setup and control of the sample environment in terms of positioning, temperature and gas environment.

11:40am **VT-TuM12 Construction and Commissioning of Tri Alpha Energy C2W machine**, ***Alan Van Drie***, Tri Alpha Energy

Tri Alpha Energy (TAE) is researching a novel fusion concept of energetic ions magnetically trapped as large orbits in a Field Reverse Configuration plasma (FRC). TAE has completed building and commissioning its latest machine, C2W.

The talk will first give a brief overview to TAE's concepts and C2W, followed by a discussion of the physics that drive the vacuum requirements, such as divertor gas loads and how we solved many of the technical vacuum challenges in order to meet our performance goals. Specifically, C2W has four $15m^3$ divertors where H_2 neutral particles from the plasma are pumped at a rate of 2,000 m^3/s. This large pumping is achieved through chemisorption onto titanium films deposited onto LN2 cooled multi-scaled surfaces. Additionally, each divertor has a $200m^3/s$ activated charcoal cryopump optimized for pumping H_2.

Vacuum Technology Division
Room: 7 & 8 - Session VT+MN-TuA

Pumping

Moderators: Tamirisa Apparao, SHI Cryogenics Group, Julia Scherschligt, NIST

2:20pm **VT+MN-TuA1 Silicon-micromachined Turbomolecular Pump**, ***Wei Yang***, PD Sciences LLC **INVITED**

Deep miniaturization of sensing and analytical instruments, such as mass spectrometers, vacuum electron devices, atomic clocks, and cold atom devices, are pushing the limit of conventional vacuum packaging technologies in micro scales. Ultra-high vacuum (UHV) of 10^{-6} to 10^{-10} torr which are beyond the capability of current passive packaging technologies, have become increasingly necessary for stable operation and high performance. Although 10^{-6} torr and higher vacuum levels are routinely achieved in macro scale systems by passive sealing and getters, maintaining such vacuum at chip-scale has unique challenges arising from scaling laws and practical limitations. Therefore, a micro scale UHV pump is highly desirable as an enabling component for a wide range of mobile or miniature instruments.

Integration of silicon MEMS and precision metal machining offers a viable path to new capabilities unattainable in their own native environments. We will present such an accomplishment in the development of a micro turbomolecular pump that takes advantage of the high-density microstructures from silicon microfabrication, and the range of motion from a precision spindle. Major achievements include compression ratio over 10^6 and maximum stall pressure of 100 Torr at relatively low tip speed of 120 m/s. This is a major milestone in the pursuit of moving UHV systems from laboratories to mobile platforms. Of particular significance, the successful demonstration of the molecular pumping against such a high exhaust pressure, a direct consequence of dimensional downscaling, points to the feasibility of a single-stage system from UHV to atmospheric pressure in miniature scales. We will discuss key technical challenges such as silicon fabrication, high-tolerance bonding, scaling analysis and simulation methodology, and touch on potential applications in small-scale thermal mechanical systems.

3:00pm **VT+MN-TuA3 A Rigorous Approach to Effluent Gas Management for the Vacuum Processing Industry**, ***Paul Dozoretz***, MKS Instruments, Inc. **INVITED**

Vacuum processing is consistently gaining momentum in the manufacturing of novel materials relying on thin film coating and implantation technologies. The vacuum systems developed for such new applications borrow from many of the standard vacuum processing techniques but consistently push the design limits in terms of the amount of precursor gas consumption and by-product mass generation. In order to handle the large amounts of effluent produced in some of these these processes it has become essential to better understand gas dynamics for the effluent flowing out of the chamber and into the pumping systems. In most cases, effluent gas must be captured or trapped before it can reach and irreversibly damage the pumps and before it can become a danger for the personnel operating the vacuum manufacturing tools. With the aid of gas dynamic modeling software, our engineering team has been able to better understand flow through effluent lines and develop more efficient gas trapping solutions for very novel applications spanning from the semiconductor to the aeronautical industries. In this presentation we describe the rigorous methodology used to guide and validate the design effluent gas handling systems. Detailed understanding of process chemistries and effluent physicochemical properties, combined with gas dynamic flow modeling, has revolutionized the way our team approaches effluent gas management and improved the speed at which customer effluent needs can be addressed.

4:20pm **VT+MN-TuA7 Compatibility of NEG Pumps with Particle-sensitive Applications: A Review of Recent Experimental Evidences**, *P. Manini, E. Maccallini*, ***Marco Urbano***, *M. Mura, T. Porcelli, F. Siviero*, SAES Getters, Italy

Non Evaporable Getter (NEG) pumps are frequently used when large pumping speeds for H_2 and active gases (i.e., H_2O, O_2, CO, CO_2) are required in conjunction with very small weight and size, reduced magnetic interference and vibration, or negligible power consumption. Thanks to these qualities NEG pumps are widespread in basic and applied research, such as particle accelerators, storage rings, synchrotrons and physics projects to achieve UHV or XHV conditions. Moreover, their use is becoming familiar in UHV analytical instrumentation such as SEM, TEM, surface science as well as portable mass spectrometry and transportation vacuum boxes.

In spite of the excellent results in terms of pressure (10^{-11} mbar are currently achieved in many machines and values lower than 10^{-12} mbar have been measured in various experiments using NEG pumps), application in cryogenic superconductive radio frequency (SRF) cavities and other particle sensitive systems is not common so far. As a matter of fact, the use of NEG pumps is limited as a precaution against potential dust emission, which can be transported inside the vacuum envelope and may interfere with the electromagnetic fields and promote unwanted quenching phenomena.

Nevertheless, these systems could greatly benefit from the high pumping speed and compactness of NEG pumps, so that an assessment of the actual risk of dust release is gradually being undertaken by different players, including potential users in the accelerator community. Here we present and compare experimental data on particle emission collected with several techniques both on compressed and sintered NEG elements, discussing the differences. In particular, a class of sintered getters based on the ZAO® alloy proved to have extremely low particle emission, as shown by tests carried out in actual SRF cavities, where no measurable particle contamination as well as detrimental effect on the cavity efficiency and performances was observed.

4:40pm **VT+MN-TuA8 NEG Coated Chambers for XHV**, ***Marcy Stutzman***, *P.A. Adderley, M. Poelker*, Thomas Jefferson National Accelerator Facility

Non-evaporable getter (NEG) thin films are typically applied to uniform diameter tubes, such as used for accelerator beamlines. We have been extending the successful application of NEG coating to larger diameter and non-uniform chambers, such as the 36 and 41 cm diameter chambers for the Jefferson Lab polarized electron source, as well as atom trap chambers for MIT and JILA. We show that by combining the NEG coating with a small ion pump to handle the non-reactive gasses, the chamber can reach the low 10^{-12} Torr range, and adding additional NEG pumping yields extreme high vacuum (XHV), with measured pressure below $8x10^{-13}$ Torr. With this demonstration of a reliable and reproducible method to achieve room temperature XHV, we hope to demonstrate the benefits of NEG coated chambers beyond accelerator physics applications to other fields of physics and materials research.

5:00pm **VT+MN-TuA9 Ion Pump Noble Gas Stability Mechanism of Titanium Cathode Material**, ***Anthony Wynohrad***, Gamma Vacuum

It has long been established that ion pumps with titanium cathodes cannot pump large quantities of noble gases without releasing them back into the vacuum environment. Argon is the typical gas chosen for study of this phenomenon due to its prevalence in atmospheric composition and tendency for use in vacuum depth profiling applications. Traditional resolutions to Ar release is through the addition of denser cathode material (Tantalum) or titanium cathode architecture manipulation (triode). Various reports have shown the long term Ar stability of these methods to be subject to manufacturer claims.

To resolve reported discrepancies of Ar stability in ion pumps with titanium and tantalum cathodes, a detailed study of titanium with various physical attributes was conducted. Five different titanium/titanium alloys were tested for Ar instability at standard depth profiling pressures rather than accelerated high pressure testing. The conclusion was reached that varying the physical properties of the titanium can cause ion pumps to become Ar stable or Ar instable. Additionally, the time to reach instability is directly in correlation to the physical attributes of titanium.

5:20pm **VT+MN-TuA10 Ricor's MicroStar/Nanostar Compact Water Vapor Cryopump: Applications and Model Overview**, ***Rodney Harris***, Ricor-USA, Inc., *I. Nachman, T. Tauber, M. Kootzenko, B. Barak, E. Aminov, D. Gover*, RICOR Cryogenic & Vacuum Systems, Israel

Ricor Systems has developed a compact, single stage cryopump that fills the gap where GM and other type cryopumps can't fit in. Stirling cycle technology is highly efficient and is the primary cryogenic technology for use in IR, SWIR, HOT FPA, and other IR detector technology in military, security, and aerospace applications.

Current GM based dual stage cryopumps have been the legacy type water vapor pumping system for more than 50 years. However, the typically large cryopanel head, compressor footprint, and power requirements make them not cost and use effective for small, tabletop evaporation / sputtering systems, portable analysis systems, load locks, and other systems requiring small volume vacuum creation from medium, high, and UHV levels. The compact NanoStar configuration was designed specifically to address this vacuum chamber size area.

This single stage cryopump works well in-line with diffusion and molecular turbopumps. Studies have shown effective cooperation with non-evaporable getter technology as well for UHV levels.

Further testing in this area are ongoing. Temperatures created by Stirling cycle cryogenic coolers develop a useful temperature range of 40 to 150K. Temperatures of approximately 100 K are sufficient to condense water and all hydrocarbons oil vapors. The wide temperature range can freeze out many other gaseous compounds.

Tuesday Evening Poster Sessions

Vacuum Technology Division
Room: Central Hall - Session VT-TuP

Vacuum Technology Poster (and Student Poster Competition)
Moderators: James Fedchak, NIST, Yevgeniy Lushtak, SAES Getters USA

VT-TuP1 Ion-Cathode Bombardment in a DC Deuterium Glow Discharge for High-Density Deuterium Cluster Formation in Metals, ***Erik Ziehm***, *G.H. Miley*, University of Illinois at Urbana-Champaign

A deuterium glow discharge is modeled to obtain angular and energy distributions of incident ions on the cathode vs plasma conditions. The model uses a DC Discharge module in COMSOL Multiphysics® [1] coupled to a Particle Tracing module while utilizing Townsend coefficients for prominent reactions derived from a Boltzmann Two-Term Approximation. Care is taken to use appropriate ionization and dissociation reaction cross sections for deuterium as these values present isotopic differences. The model is benchmarked by Langmuir probe measurements of the electron energy distribution along the cathode dark space and negative glow regions. This model is then used to determine the effects of ion incident energies and dose on the creation of the high-density clusters of deuterium atoms beneath the cathode surface layer. Temperature Desorption Spectroscopy (TDS) complimented with X-Ray Diffraction (XRD) is employed to determine the clusters' trapping energies and densities.

VT-TuP2 Low-cost Device Fabrication and Vacuum Packaging for Energy Efficient Field Emission Lighting, ***Sushma Shrinivasan***, *C.E. Hunt*, University of California - Davis

With the growing emphasis on climate change and global warming and the resulting need to cut down energy usage, energy-efficient lighting technologies that resemble day light spectrum and do not present health hazards are extremely attractive. Field emission lamps (FELs) have been presented as a viable alternative to existing lighting technologies with several advantages including (i) spectrum similar to daylight (ii) environment-friendly (iii) no health hazards to name a few. In this regard, the primary goal of this poster is to present a low-cost, simple device fabrication technique for a typical FEL. The FEL device consists of a base plate, face plate and side-wall (all made out of glass). The glass package is built by attaching the various components using ultra high vacuum epoxy. The base plate and face plate comprise of the cathode (reticulated vitreous carbon) and anode (aluminum coating) respectively with the face plate additionally comprising of a phospor coating. The exhaust tube for the device is located on the side-wall. The device is then attached to a turbomolecular pump and pumped down to vacuum levels of 1E-6 Torr. This level of vacuum is shown to activate the barium getter that is attached to the baseplate before the device packaging. The activation of the barium getter is performed using an in-house induction coil and a radio frequency generator operated at a frequency of 300 kHz. The packaged device when combined with a high voltage DC power supply is anticipated to lead to a low-cost energy efficient lighting option that has a spectrum similar to incandescent lamps with an energy consumption comparable to compact fluorescent lamps.

VT-TuP3 High Precision Measurement Of Tube Conductance From Pressure Decay Curve, ***Tim Verbovšek***, *B. Šetina Batič, J. Šetina*, Institute of Metals and Technology, Slovenia

A unique vacuum system for precise measurement of gas throughput through a tube connecting a vacuum chamber and a pump was constructed. A conductance of a duct between the pump and the vacuum chamber determines the rate of pressure decay $p=p(t)$ when nonadsorbing gas is pumped. If gas back-streaming from the pump through the duct is negligible, and gas temperature in the duct is the same as in the chamber, the conductance C of the duct can be calculated from the time derivative of the logarithm of the pressure decay by $C=V\times\mathrm{d}(\ln(p))/\mathrm{d}t$, where V is volume of the chamber. The simplicity of this equation is also a basis for a very high precision of such measurement. Uncertainties which have to be considered are related to the volume of the chamber and deviations from isothermal conditions. Since logarithm of the measured $p(t)$ curve is used to calculate conductance, any correction factor of the vacuum gauge cancels out. Moreover, different sensitivity of the vacuum gauge for different gases is totally unimportant, so only random noise of the measured pressure contributes to the uncertainty. Estimated relative uncertainty of measured conductance is less than 0.3%. Reproducibility (with volume V unchanged) is even less than 0.2%.

Very high precision of this method enables studies of the influence of gas-surface interaction on the tube conductance in molecular regime. Any changes of tangential momentum accommodation coefficient reflect in variation of the tube conductance. We will present results of measurements of conductance of a long stainless steel tube with inner diameter of 7.76 mm for initial state and after different treatments (all at 300 °C for 24 h): exposure to O_2 at 0.1 Pa, vacuum bake, and exposure to H_2 at 0.1 Pa. Conductance was measured for gasses He, Ne, Ar, Kr, CH_4 and N_2 in the range of Knudsen numbers from 0.01 to 1000. Variations of molecular scattering on the tube surface resulted in changes of tube conductance of more than 10 % for He, while for N_2 and CH_4 the observed changes were less than 2 %.

VT-TuP4 Using a High Vacuum Equipment Trainer (HVET) System for Hands-on Learning, ***Del Smith***, *N. Louwagie*, Normandale Community College

In keeping with the theme of the 2017 Symposium, "Surfaces, Interfaces and Materials: A New Vision," this paper will discuss the experiences of Normandale Community College (Bloomington, Minn.) instructors offering academic courses in vacuum and thin film technology via a telepresence interface. We believe this new model, which brings together on-campus and remote learners in real time, is a positive direction for technical education. Over the past two years, several organizations throughout the U.S. have enrolled their employees and students in courses, which emphasize hands-on learning with a vacuum trainer system.

Normandale staff in the Vacuum and Thin Film Technology Program, with input from senior technicians from several Minnesota-based industries, designed a High Vacuum Equipment Trainer (HVET) system to use in the classroom. The HVET system has $5x10^{-6}$Torr base pressure capability and supports demonstrations of vacuum technology operations such as gauging, gas sensitivity, leak testing, RGA analysis, and plasma generation. Students use the HVET system to practice pumpdown sequences, pumpdown curves, rates of rise, and conductance in a lab environment. There are four copies of the HVET, which can be disassembled and shipped to participating sites; students assemble the HVET system as part of their initial learning experience.

The telepresence classroom at Normandale was designed with a combination of high-performance audio and video feeds and multiple monitors to show close-ups of lab experiments and to allow the instructor to respond to nonverbal cues from remote students. Normandale offers an AAS degree in Vacuum and Thin Film Technology. Two courses from this program are available via telepresence: Intro to Vacuum Tech and Vacuum Analysis and Troubleshooting. Eventually, two more courses will be brought online: Thin Film Deposition; and, Foundations in Vacuum Technology, which will teach chemistry and math concepts in the context of vacuum science. Students will be able to complete these courses in a year, via telepresence, earning a Vacuum Technology Certificate and 12 academic credits, which are applicable towards the AAS degree. This paper will discuss how the trainer systems and telepresence provide remote students with access to formal education in vacuum and thin film technology. (This work was made possible in part by a grant from the National Science Foundation: DUE #1400408.)

VT-TuP5 Advanced Metal Sealing Solutions for Critical Industry Applications, ***Ryan McCall***, Technetics Group

As operating conditions in many critical industries, such as vacuum and semiconductor, continue to face increasingly extreme temperatures and pressures, sealing technologies are facing challenges that cannot be met with traditional materials and methods. Additionally, there are growing pressures to ensure the protection of the environment remains a top priority for companies and that they are consistently meeting the strict requirements associated with international regulations. More and more manufacturers are turning to metal seals as their solution of choice as today's industry requirements are becoming more and more stringent.

Because of these stringent requirements, metal seals designed for use in modern sealing applications must be resilient to the extreme temperatures and pressures that they are exposed to in a number of harsh environments. Seal designs must also allow for bi-direction flow, be suitable for axial and radial applications, provide a long sealing life, be resistant to corrosion, be able to remain as leak tight as possible under semi-dynamic motions and account for thermal expansions.

This presentation will explain the factors to consider when selecting a metal sealing solution and what types of seal designs (spring-energized, O-rings, C-rings, E-rings, etc) are best suited for each given aspect of a critical industry application. The factors that will be discussed in the presentation will include but are not limited to seating load, seal function, seal material and thickness, required leak rate, coatings, controlled compression and

surface finish. Additionally, the specific applications where metal seals are most suited and why will also be discussed.

VT-TuP6 Development of the Residual Gas Analysis for Large Air Tight Packages, ***Yusuke Nishikawa***, Advanced Technology R&D Center Mitsubishi Electric Corp., Japan, *M. Kinugawa*, Advanced Technology R&D Center Mitsubishi Electric Corp.

A hollow airtight packaging structure is used for electric / electronic parts (e.g. lamps, high frequency devices, etc.) to improve reliability and securing characteristics. It is crucial to know the variety and amount of impurities in the structure's internal gas, which adversely affects said properties. We have developed a gas analysis technique for small parts with an internal volume of 1 cm3 or less [1].

In the case of small package devices, it is possible to break it in a vacuum chamber and analyze the gas inside. However, when analyzing residual gas with a large package, it is very difficult to prepare a larger chamber and break the sample. Furthermore, if the surface area in the package is large, the influence of desorption of adsorbed molecules cannot be ignored. It is difficult to accurately measure the pressure of residual gas.

In this study, we present a new technique for analyzing gas in sealing devices that can be flexibly applied and can minimize the influence on the state of the residual gas composition even for various large samples.

The testing apparatus consists of a gas sampling chamber and an analysis chamber, with both connected to a vacuum valve and an exchangeable orifice. The analysis chamber has a quadrupole mass spectrometer and is exhausted continuously by a turbomolecular pump. The gas sampling chamber has a perforator, a vacuum gauge, and an additional pumping system. The volume of the gas sampling chamber is sufficiently small relative to the volume of the sample. The package sample is connected directly to the connection port of the gas sampling chamber. By using a small gas sampling chamber, an external connection of the sample, and a variable orifice, this technique can be flexibly applied to various large samples.

We measured the temperature of the internal pressure of a certain package sample. As a result, we found that the pressure increase was five times higher than when considering by the ideal gas law and ion intensity of water was increased. We estimated them as the adsorption and desorption levels of gas on the surface of the internal components.

To further advance this technique, we found that it is important to know the influence of gas adsorption and desorption inside the package. We will research the gas adsorption and desorption for the component parts affecting residual gas partial pressure.

[1] M. Kinugawa, et al.: Mitsubishi Denki giho Vol. 81 No. 3 231 (2007).

VT-TuP7 ARIEL RIB Transport line Vacuum System, ***Geoffrey Hodgson***, TRIUMF, Canada

The vacuum system of the Radioactive Isotope Beam (RIB) transport line is part of the Advanced Rare IsotopE Laboratory (ARIEL) at TRIUMF. This beam line will accept three simultaneous RIBs and transmit two of them to low energy experimental facilities and one to further accelerators. The RIBs will consist of ions of exotic radioactive species with masses ranging from 6 to 238 amu and energies from 10 to 60 keV. RIBs will be extracted from two new ARIEL target stations and one of two existing ISAC target stations. In ISAC, and one of the ARIEL target stations, targets materials ranging from metals, oxides, carbides to actinide compounds are irradiate by 50kW, 500 MeV protons from TRIUMF's main cyclotron. The second ARIEL target will make use of 100 kW, 35 to 50 MeV electron beam which is converted to high-energy gamma rays in a thin layer of gold. These gamma rays are used for photodisintegration of beryllium oxide or photo photofission of uranium carbide. The beam line will be built on two floors. The lower floor will contain Medium and High Resolution mass Spectrometers (MRS and HRS) having 1:5,000 and 1:20,000 resolution, respectively. The upper floor will contain charge breeding equipment to provide a typical mass to charge ratio of 7 to allow further acceleration of heavier isotopes. The design pressure is $3 \cdot 10^{-8}$ Torr for singly charged beams and $1 \cdot 10^{-8}$ Torr for highly charged ion beams based on beam loss calculations. The system will use turbo pumps and scroll pumps to achieve the vacuum. Individual components of beam line and beam steering equipment were tested to determine their conductance in molecular flow, and the model of the vacuum profile was created. A 14.5 m prototype section of beam line was built and used to validate the profile model. The beam line is divided into isolable sections, and each section will have a standard vacuum pumping station to facilitate controls and interlocks.

VT-TuP8 Operational Regime of 2 million L/s Cryobox Pump on Tri Alpha Energy's C2W Machine, ***Ernesto Barraza-Valdez***, *A. Van Drie*, Tri Alpha Energy, Inc.

Tri Alpha Energy requires a large pumping speed of approximately 2 million L/s for hydrogen and 1.5 million L/s for deuterium in each of the four divertors (22,000 L) on C2W. To accomplish this TAE has developed a liquid nitrogen cooled, titanium getter pump. The experimental pump speed was tested for a variety of parameters including purity of titanium (99.995% and 99.97% Ti), various temperatures, and saturation. Purity of titanium showed no effect on the pump speed but there was a clear correlation with the temperature and saturation. With this, TAE had discovered a regime of operation during plasma shots in C2W.

Vacuum Technology Division
Room: 7 & 8 - Session VT-WeM

Transfer and Ultraclean Systems, Particle Control, and History

Moderators: Jason Alfrey, Vacuum Technology, Inc., Marcy Stutzman, Thomas Jefferson National Accelerator Facility

8:00am **VT-WeM1 Applications and Challenges of UHV- and Cryo Transfer of Samples Between Independent Analytical Systems**, ***Urs Maier***, *S.A. Köster, D. von Gunten*, Ferrovac GmbH, Switzerland, *S. Yoshizawa, T. Uchihashi*, National Institute for Materials Science, Japan, *S. Rauschenbach*, Max-Planck-Institute for Solid State Research, Germany **INVITED**

To answer demanding questions in the characterization and analysis of sample surfaces a variety of high performance atomically precise methods are available. Molecular beam epitaxy (MBE), focused ion beams (FIB), electrospray ion beam deposition (ES-IBD) or even vitrification of biological samples can fabricate surfaces of great complexity with atomic/molecular precision. Similarly, analytical methods like scanning probe microscopy (SPM), electron microscopy (SEM/TEM), photo- or electron spectroscopy (e.g. XPS), low energy electron holography or secondary ion mass spectrometry (SIMS) provide spatial or chemical insight of highest precision. A combination of these methods is often desirable while these methods require a clean environment, typically ultrahigh vacuum (UHV, 10^{-10} mbar) and sometimes cryogenic conditions to maintain the sample in a state that allows for meaningful results.

A sample transfer with undisrupted vacuum and cryogenic conditions cannot always be achieved via a direct linkage between the instruments. More complex combinations, involving many different methods, are often impractical. The ability to transfer samples under well controlled environmental (UHV) and thermal conditions between independent analytical systems therefore greatly extends possible applications of the instrumentation.

Here we present the NexGeneration UHV suitcase system that allows to transport samples in UHV environment between instruments of even far separated facilities. The system consists of a lightweight and readily transportable vacuum chamber (the suitcase) which is actively pumped by a combination of a getter/ion pump (SAES, NEXTorr) using a battery powered controller which allow for up to 2 days off-grid operation. The transfer is facilitated via loadlocks attachable to arbitrary systems. The suitcase is of modular configuration allowing the addition of sample storage positions, adaptation to different carriers, as well as choosing from different types of sample transporters to handle the sample. Above all, a cryogenic variant of the suitcase has recently been developed, in which samples are actively cooled down to -190°C on a liquid nitrogen cooled stage situated within a thermal shield.

In the presentation we show our implementation of a sample transfer with the NexGeneration UHV suitcase system and show configurations established for different research groups. We further present data measured from samples transported within our suitcase that shows the cleanliness expected from samples that were maintained in UHV, clearly showing the capability of our system for a large variety of applications.

8:40am **VT-WeM3 Ultra-clean Sample Transportation in an EUV Exposure System**, ***Freek Molkenboer***, *N.B. Koster, A.F. Deutz, B.A.H. Nijland, P.J. Kerkhof, P.M. Muilwijk, B.W. Oostdijck, J. Westerhout, C.L. Hollemans, W.F.W. Mulckhuyse, M. van Putten, P. van der Wall, A.M. Hoogstrate, J.R.H. Diesveld, A. Abutan*, TNO, Netherlands

In 2015 TNO started the design of a new Extreme Ultra-Violet (EUV) exposure facility, called EUV Beam line 2 (EBL2). EBL2 will be a publicly accessible test facility for EUV lithography related research and qualification. The realisation of the EBL2 started at the end of Q1 2016. On December 7th 2016 the important milestone " First light" was accomplished.

EBL2 is designed to be able to load a wide range of sample types, including the EUV industry standard 6" reticles. To achieve this, all the samples are loaded using the SEMI standardised EUV dual pods.

Sample loading for the EBL2 facility starts at the Atmospheric Handler. The EUV dual pod is opened, and the Atmospheric Handler robot transfers the sample to the load lock of the EBL2 system. The Atmospheric Handler has several ultra-clean environments to limit the particle contamination on the samples.

After the load lock is evacuated to vacuum, the robot of the Vacuum Handler will transport the sample to the operator-selected module of the EBL2 system. Besides the load lock, the Vacuum Handler connects to the Expose Chamber, an XPS, and two chambers that are used for storage and cleaning of samples. In the Exposure Chamber samples can be exposed to EUV irradiation in various controllable gas environments. An XPS is available for surfaces analysis after an experiment while maintaining vacuum. The handling between the chambers is fully automated with multiple checks to ensure sample safety.

When a sample is transported to the Exposure Chamber the sample must be flipped from a horizontal loading position to a vertical mounting position. This is due to the design constraints of the EUV source and illumination module of the EBL2 system. After this flip from horizontal to vertical the sample must be positioned and clamped against the cooled Sample Chuck.

Both the flip and the clamping motions use pneumatic actuated bellows that are located inside the Exposure Chamber.

The Sample Chuck positions the sample in the EUV irradiation spot across the entire 6" reticle. The movement of the Sample Chuck is accomplished with a Hexapod that is located outside the vacuum. The vacuum barrier between the hexapod and the Sample Chuck is a large edge welded bellow.

During this presentation we will discuss our implemented design solutions for sample handling in ultra-clean vacuum. The objective of the design and implementation are to maintain the stringent vacuum and particle requirements for these kind of experiments.

9:00am **VT-WeM4 Oxidation and Contamination Monitoring Methods for Air Sensitive Materials Transfer: From Glove Box to UHV Surface Analysis**, ***Hugo Celio***, *K.B. Ohlinger*, University of Texas at Austin

The performance of lithium ion-batteries is steadily improving but there is still need for higher energy density and cycle life in consumer applications. An ex situ investigation of the composition is crucial for investigating performance issues. After a cycling period, the cathode (or anode) material is extracted from a battery coin cell under an argon environment of a glove box. However, a glove box also contains traces of O_2 and H_2O in the 1-part-per-million range, an unknown amount of adventitious hydrocarbons and inorganic impurities. These traces of oxidants and contaminants are carried along as battery materials are transferred from a glove box to a UHV chamber for surface analysis.

An interface designed to transfer air sensitive materials from a glove box to an ultra-high vacuum (UHV) chamber for surface analysis was previously presented[1]. This interface is called an interface for pressure-to-vacuum environmental sample transfer, or IP-VEST. It is coupled to a UHV chamber equipped with X-ray photoelectron spectroscopy (XPS). The IP-VEST has a built-in method for transfer reliability. However, there is no known method to monitor the degree of oxidation to air sensitive materials from exposure to trace levels of oxidants in argon.

We selected silicon, silver, tin and lithium as a set of reference materials to monitor their surface oxidation and contamination from exposure to trace levels of oxidants, organic, and inorganic contaminants during the environmental transfer of air sensitive battery materials as described above. The preparation procedure for the reference materials is a simple mechanical step which is carried out in the glove box. We focused on evaluating the oxidation rates of clean Si, Ag, Sn and Li with respect to travel time, which is dictated by distance between the glove box and the IP-VEST/UHV chamber. At UT, five glove boxes are located within short distances of each other, entailing a preparation and travel time of less than 30 minutes. Upon arrival to the UHV chamber, the capsule, containing the air sensitive battery materials and reference materials, is coupled to the load-lock of the IP-VEST. This transfer process requires 1 hr. for samples from atmospheric argon pressure to high vacuum conditions. Based on XPS data, the surface oxidation of the reference materials significantly varies but yield a range of oxidation rates. These oxidation rates can be compared to the oxidation rates of some battery materials. The environment of the glove boxes is effectively inert for most cathode materials but insufficient for highly reactive battery materials like lithium.

[1] AVS 2015 and U.S. Patent Application Serial No. 14/445,650

9:20am **VT-WeM5 Particle Contamination Control in the Accelerator Vacuum Systems of the European XFEL**, ***Lutz Lilje***, *S. Lederer*, DESY, Germany **INVITED**

For the European XFEL accelerator vacuum about 1,5 km of vacuum system have been assembled with procedures that result in a low particulate contamination inside of the vacuum components. For this mechanical design aspects as well as cleaning and installation procedures needed to be adapted to a large variety of beam line components. An example is the development

of dedicated clean rooms for the installation of components in the accelerator tunnel.

The experience with the installation is presented as well as preliminary results from the commisioning of the accelerator. An initial correlation of difficulties observed during the installation process with performance of modules will be discussed.

11:00am **VT-WeM10 Development, Solution of Design Issues, Final Design and Performance of an Electrostatic Triode Getter-Ion Pump, 1967-1973**, ***Paul Arnold***, MKS Instruments, Inc. **INVITED**

History of a completed commercial electrostatic triode getter-ion pump, where ion pumping and getter pumping were separated allowing preservation of the getter at UHV, will be presented. Also covered will be the solutions of combining high temperature, high voltage, high getter sublimation rate, and ultra-high vacuum in one pump design from the late 1960s. The getter was operated with active gas pumping speed as a direct function of the power to the getter and was independent of the ion pumping, allowing preservation of the getter material at UHV while maintaining full ion pumping speed. The physical electronics of a hot filament ion pump design with four pumping cells, each with dual filaments, will be shown. The successful joining and assembly of many refractory materials, some at temperatures reaching 1600 Celsius, will be described. The pump operates with electrodes at voltages up to 4000 volts in the environment of a titanium sublimation rate up to 0.02 grams per hour while maintaining adequate resistance values of the many insulators. Pumping speeds for various gasses will be displayed, along with pumpdown curves for many gasses with a gas type comparison to a sputter-ion pump from the same 1960s era. Automatic turn-on and turn-off pressure indications were provided by a heat-loss gauge with full-scale resolution of 10 millitorr. This Invited Talk is part of the AVS History Committee's endeavor to preserve and promote our vacuum technology history.

11:40am **VT-WeM12 The Modern View of the Vacuum**, ***H. Frederick Dylla***, American Institute of Physics

The concept of the vacuum has evolved from ancient to modern times. Ancient Greeks did not believe in the concept of vacuum-empty space in which nothing exists. With their early formulation of atom-like particles, they believed that matter was completely space filling. As civilization moved into the "Enlightenment" and the early industrial age, a practical definition of vacuum became any space evacuated to a pressure less than ambient. This is still a practical definition of vacuum in contemporary times, where state-of-the-art techniques can produce extreme vacuum levels-approaching matter densities of less than a molecule/cm^3. However, from the standpoint of contemporary physics, we have moved back to a view that the vacuum is not empty space devoid of content. Paul Dirac's theory of Quantum Electrodynamics, the most precisely experimentally benchmarked theory in science, portrays empty space as being filled with quantum fluctuations: virtual particle-antiparticle pairs appearing and disappearing on extremely short (Planck) time scales. The present status of cosmology research adds additional complexity to the concept of a perfect vacuum. Quantum fluctuations underpin Alan Guth's inflationary model of the universe's expansion following the primordial Big Bang. His widely accepted analysis explains the high uniformity of matter density in the observable universe-a part in 10^4. Quantum fluctuations in space drove an immense (10^{28}) expansion of the primordial universe using the latent energy in a so-called false vacuum. Over the last two decades additional observations of the universe's expansion rate, have shown that the visible components of the universe (matter and radiation) account for only about 1% of the content- 30% resides in dark matter and 70% in dark energy. Characterizing these latter two components remains on the forefront of modern physics research, and clearly a perfect vacuum is far from empty.

12:00pm **VT-WeM13 History of Very Thick Film and Bulk Sample Group IIIB, IVB, VB and Rare Earth Materials for Various Vacuum Applications**, ***James L. Provo***, J.l. Provo Consulting

History of Very Thick Film and Bulk Sample Group IIIB, IVB and Rare Earth

Materials for Various Vacuum Applications

James L. Provo [a]

Consultant, J.L. Provo Consulting, Trinity, FL 34655-7179

Thick occluder films of hydride materials are extremely hard to produce without

flaking or cracking. This paper discusses methods of how to prepare thick films

and bulk samples (i.e., rods and wires) for many applications including accelerator

research for cancer therapy, intense neutron source, and particle-beam fusion

diagnostic beam focusing studies. These thick films ~ (≥ 5,000 to 15,000 nm thick) of various hydrides are sensitive to oxidation and are easily contaminated by

improper handling. They must be specially prepared to reduce internal stresses due

to temperature variations during processing, and stresses due to hydriding and to

substrate configuration (i.e., curved surfaces). This paper will discuss techniques

developed at the General Electric Neutron Devices Department (GEND), in Largo, FL,

in the mid-1970's to the late 1990's to produce stress free and thus flaking and crack

free samples of thick films and bulk samples. Items studied include, Er, Sc, and Ti

thick film hydrides on a Cr underlay, on various substrates, bulk rod samples (0.635

cm O.D. by 2.54 cm long) for basic material heat capacity and thermal diffusivity

studies as a function of hydride loading, Nb and V wires 10 and 20 mil O.D. by 5.08

cm long in bundles of ~30 wires for neutron vibration spectra studies and 20 mil O.D.

by 1.27 cm long Ti wires for mass spectrometer calibration studies. Film samples were

prepared by standard E-beam evaporation techniques and then non air-exposure loaded.

Bulk samples were cleaned, weighed, and then loaded with a Sievert's precise gas

quantity loading system. Special processing to accomplish flake and crack free samples,

included heating sample substrates for thick films to 450°C, evaporating at a controlled

rate of 10 nm/min.,which takes ~ 8.3 hrs. for 5,000 nm films and ~25 hrs. for 15,000

nm films, followed by non-exposure loading (i.e., leaking D_2 or T_2 gas into the loader) at a

rate of 1Torr/hr. until 50 Torr is reached, which will take ~2-days, holding at temperature

and pressure for ~ 8hrs, then cooling down at a rate of 1°C/ min.from 450°C to room

temperature. Er films are cooled to ~ 320°C, then gas is removed to the source bed to

prevent trihydride formation, before cooling to ~ (25°), which will take ~ 7.5hrs. Using

the process described, very successful results were obtained.

[*] Formerly, Principle Member of the Technical Staff at Sandia National Laboratories,

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Vacuum Technology Division
Room: 20 - Session VT-WeA

The History and Future of Materials, Surfaces and Interfaces (ALL INVITED SESSION)

Moderators: Gregory Exharos, Pacific Northwest National Laboratory, Amy Walker, University of Texas at Dallas

2:20pm **VT-WeA1 The 14-billion Year History of the Universe Leading to Modern Materials Science**, ***Joe Greene***, University of Illinois **INVITED**

The story of our universe begins approximately 13.8 billion years ago with the Big Bang. Many of the formative events occurred in the first tiny fractions of a second (the universe evolved from consisting of a quark/gluon plasma to form the first protons and neutrons) to minutes (free neutrons decay to electrons and neutrinos) to a few tens of thousands of years (elementary particles form the first elements, which leads to the development of stars due to local density fluctuations). Planet Earth nucleated and began to accrete interstellar debris ~4.5 billion years ago. While the lighter metal elements on earth formed primarily due to stellar supernovae explosions, the primary mechanism leading to the formation of the heavier elements has only recently been demonstrated. The first known sophisticated stone tools used by hominids date to 2.6 million years ago.

Gold is likely the first metal discovered by man, >11,000 years ago. However, unlike copper (~9000 BC), bronze (~5000 BC), and cast iron (~600 BC), it was too soft for fabrication of tools and weapons. Instead, gold was used for decoration, religious artifacts, and commerce. The earliest high-purity Au artifacts derive from NE Bulgaria ~6500 y ago; however, the largest known concentration of ancient gold mines is in the Egyptian Eastern Desert. Copper extraction from ore was already being carried out in the Balkans (E Serbia and S Bulgaria) ~7500 years ago. Spectacular copper sculptures displaying very high levels of metallurgical and artistic craftsmanship have been found in Mesopotamia (S Iraq).

Gold brazing of metal parts was first reported ~3400 BC in Sumaria. The earliest documented thin films were gold layers, some < 1000 Å thick, produced by Egyptians ~5000 years ago. Examples, gilded on copper and bronze statues and artifacts (requiring sophisticated compositionally-graded interfacial adhesion layers), were found in pyramids dating to ~2650 BC. Electroless gold and silver plating was developed much later by the Moche Indians of Peru in ~100 BC.

Early biomaterials, used as human prosthetics following successful amputations, date to 950 BC in Egypt; while the first nano-based devices, exhibiting spectacular dichroic effects due to ~200-Å-diameter Au quantum dots, were synthesized in Rome ~350 AD.

Vapor-phase deposition of thin films required the invention of vacuum pumps (~1650 through 1865). The fascinating development of crystallography begins with Plato in 360 BC.

While an historical road map tracing the progress of materials technology is interesting in itself, the stories behind these developments are even more remarkable and provide insight into the evolution of scientific reasoning.

4:20pm **VT-WeA7 Controlling Microorganisms with Bio-inspired Materials**, ***Caitlin Howell***, University of Maine

Nature was the first to create adaptive, multi-functional materials, surfaces, and interfaces. Now, the concept of borrowing ideas from Nature is one of the major drivers in the future of materials- and surface-based technology. In keeping with this trajectory, our group designs surfaces to understand and control microorganisms using bio-inspired concepts. One major application is in the control of bacterial adhesion leading to biofilm formation, which causes a wide range of problems in industry and medicine. Using a method inspired by the *Nepenthes* pitcher plant, which uses a thin immobilized layer of water to repel insects, we can create selective patterns of bacterial adhesion using common laboratory materials and simple bench-top surface treatments. Inspired by the vascular systems of plants and animals, we can then make these surfaces continuously self-replenishing by embedding channels within the material itself. The channels are then filled with excess liquid, which can diffuse to the surface and heal depleted or damaged areas. Finally, we are working to develop these materials on paper substrates to create low-cost, lightweight pathogen-handling materials for use in diagnostics or analytics. Through this work, we aim to develop new and versatile tools for the exploration and control of microorganisms.

4:40pm **VT-WeA8 Comparison of Oxygen Adsorption and Absorption on Rhodium, Silver, and Stepped Platinum Surfaces**, ***Daniel Killelea***, *R.G. Farber, M.E. Turano,* Loyola University Chicago, *E.V. Iski,* University of Tulsa, *L.B.F. Juurlink,* Leiden Institute of Chemistry, The Netherlands, *J. Derouin,* Loyola University Chicago

The interaction of oxygen with the surfaces of catalytically active transition metals has attracted much interest because of the relevance to heterogeneous catalysis. Recently, we have shown that oxygen coverages in excess of 1 ML are achievable using gas-phase atomic oxygen (AO) to dose the metal surfaces. This talk will discuss some recent results comparing the uptake of AO and O_2 on Ag(111), Rh(111), and stepped Pt surfaces. On Pt surfaces, the geometry of the monoatomic steps determines whether or not low temperature dissociative chemisorption of O_2 will occur. In addition, on Pt(553), prolonged exposure to AO does not result in O coverages in excess of a monolayer, suggesting the defects are not effective at promoting the formation of subsurface oxygen. Conversely, on Rh(111), subsurface oxygen readily forms from exposure to AO. Finally, the uptake of oxygen on Ag(111) is discussed; unlike Pt(553) or Rh(111), where little surface reconstruction occurs, Ag(111) undergoes several phase transformations as the oxygen coverage is increased. These results using AO demonstrate that UHV-compatible dosing can prepare the same surfaces resulting high pressure O_2 exposures, allowing for quantitative and structural analysis of the oxidized surfaces.

5:00pm **VT-WeA9 Single Asperity Contact and Sliding**, ***Ashlie Martini***, University of California Merced

Abstract: Nanoscale probes are widely used for surface and material characterization as well as for emerging nanoscale manufacturing techniques; they also are model single asperities and so provide a means of studying contact and relative motion between surfaces at a fundamental level. The challenge in understanding such phenomena is that experimentally-observed properties are determined by processes that occur within the interface between two materials. As such, it is desirable to complement experiments with simulations that can provide insight into the atomic-scale mechanisms within that buried interface. However, there are challenges to modeling nanoscale probe-based experiments with sufficient accuracy that the simulations can be used to explain experimental observables. We have addressed this issue in recent work by designing models to reproduce specific experiments in which nanoscale probes are used to characterize contact, friction and wear. We focus on tractable problems for which optimally-matched simulations can be directly validated by comparison to experiments and the simulations can in turn provide insight into the fundamental mechanisms underlying contact and sliding at the nanoscale.

5:20pm **VT-WeA10 Structure of Sub-nm Oxides Synthesized by Atomic Layer Deposition: From Isolated Cations to the Emergence of Crystallinity**, ***Angel Yanguas-Gil***, Argonne National Laboratory

The history of the AVS has witnessed a dramatic reduction in thickness of thin film materials. Characteristic dimensions of the order of a nanometer are commonplace for a wide range of applications, from energy storage to semiconductor processing, nanostructured photovoltaics, catalysis or beyond Von Neumann computing architectures. This has greatly reduced our margin of error in terms of achieving the right microstructure and properties as well as the long-term stability of materials, particularly when the synthesis takes place at low temperatures and the mobility of surface species is low.

Atomic layer deposition is a thin film technique that is well known for its ability to coat high surface area materials, but it also allows us to grow materials with extremely high precision and high reproducibility. This makes it an ideal model system to understand some of the fundamental aspects of the growth of materials at the nanometer scale and their stability with time and under extreme environments. In this talk I will show how through the combination of different in-situ techniques, from PDF to XAFS and FTIR, the ability to modulate surface reactivity, simulation and theory, we can study the evolution of the structure of materials from isolated cations to a bulk-like structure and isolate the main factors driving the evolution of microstructure. Looking into the future, the development of new in-situ characterization tools at synchrotron radiation facilities worldwide is going to be an enabling capability that will help us understand the driving forces behind the emergence of crystallinity at low temperatures.

5:40pm **VT-WeA11 The Power of Atomic Layer Deposition – Moving Beyond Amorphous Films**, ***Virginia Wheeler***, *A.C. Kozen, B.P. Downey, M. Currie, N. Nepal*, U.S. Naval Research Laboratory, *L.O. Nyakiti*, Texas A&M University, *D.J. Meyer, D.R. Boris, S.G. Walton, C.R. Eddy, Jr.*, U.S. Naval Research Laboratory

Atomic layer deposition (ALD) has emerged as a powerful technique to produce a wide variety of thin film materials including metal oxides, nitrides, and metals for use in numerous applications. This method has become increasingly useful as device dimensions are reduced and complexity is increased often resulting in non-planar architectures. The sequential, self-limiting reactions that define ALD enable excellent conformality on high-aspect ratio structures, angstrom level thickness control, and tunable film compositions. Additionally, ALD is conducted at low growth temperatures (Tg) which allows for integration of dissimilar materials as well as the ability to access new regions of phase diagrams in complex systems (ie. metastable phases, miscibility gaps, etc.). Traditionally, this low Tg yields amorphous films. In many applications, it is becoming increasingly advantageous to incorporate thin, conformal crystalline materials which are currently limited by the low Tg in ALD. To overcome this barrier, many have investigated post-deposition processing or plasma enhanced ALD. In this work, we will explore the advantages and limitations of approaches towards attaining crystalline ALD films through the following case studies: high quality phase transitions in ALD VO_2 and phase control of heteroepitaxial Ga_2O_3.

VO_2 is a thermochromic material that undergoes a crystalline phase change at critical temperature (68° C) resulting in drastic changes in optical and electrical properties. While crystallized ALD VO_2 films have been shown to have sufficient transitions, they are deposited amorphously. Crystalline films are only obtained through a post-deposition anneal (500-600° C in O_2) since the vanadium precursor degrades at elevated temperatures (>150° C). However, this high temperature anneal limits the integration of ALD VO_2 films with other materials and without careful consideration of anneal parameters such as temperature, pressure and gas environment can alter the stoichiometry and structure of the initial ALD VO_2 film.

A plasma enhanced ALD (PE-ALD) process was used to attain heteroepitaxial Ga_2O_3 films on c-plane sapphire substrates at 350° C. This is about half of traditional CVD or MBE methods, showing the benefits of PE-ALD. Furthermore, the crystallinity and phase composition of the Ga_2O_3 film can be control with growth temperature, plasma gas flow, and pressure. For example, reducing the chamber pressure an order of magnitude resulted in a shift from pure β-Ga_2O_3 to pure α -Ga_2O_3 at low pressures. Initial results correlating plasma species with phase control will be presented and discussed as a way to overcome the limitations of the low ALD Tg.

6:00pm **VT-WeA12 The Cathodic Arc Plasma from Multi-Element Cathodes**, ***Robert Franz***, Montanuniversität Leoben, Austria

Cathodic arc deposition has been established as one of the standard techniques for the physical vapour deposition of thin films and coatings as it allows the synthesis of a wide variety of materials including metallic films, but also nitrides, carbides and oxides if a reactive background gas is used. In addition, the highly ionised plasma and the achievable high deposition rates allow a variety of control mechanisms to influence the film growth while the manufacturing costs remain rather low due to the short deposition times. With the advent of multifunctional thin films and coatings, the use of multi-element cathodes providing the non-gaseous elements during the synthesis has become an industrial standard. However, a detailed understanding of the discharge properties is vital for the further optimisation of the deposition processes to enable synthesising thin films or coatings with improved properties.

In the present work, the cathodic arc plasma from CrAl and NbAl composite cathodes is studied in detail. The ion energies are measured element, charge state and time resolved in vacuum conditions as well as in the presence of an inert and reactive background gas. Differences in the ion energy and charge state distributions between the plasma from the composite cathodes and corresponding single-element cathodes are noticed. Since it is expected that such differences originate from changes on the cathode surface, namely the formation of intermetallic phases, the erosion behaviour of the cathodes is also analysed. The latter is supported by density functional theory calculations providing additional information about the cohesive energy of the elements which includes the influence of surface texture and temperature. The presence of a background gas generally leads to a reduction of ion energies and charge states. Cathode poisoning effects are mainly observed outside of the main erosion zone. All obtained data are discussed on the basis of correlating the material properties of the cathodes and the plasma properties of the established discharges in order to obtain a comprehensive understanding of the cathodic arc discharge from multi-element cathode and to guide the further development of the deposition of thin films and coatings using such discharges.

Tribology Focus Topic
Room: 10 - Session TR+AC+TF+VT-ThM

Lubricant, Coatings, and Biotribology

Moderator: J. David Schall, Oakland University

8:00am **TR+AC+TF+VT-ThM1 Superlubricity of Hard Compliant Carbon Coatings with Green Lubricants: Role of Surface Chemistry and Structural Changes**, ***Maria-Isabel De Barros Bouchet***, Ecole Centrale de Lyon - LTDS, France **INVITED**

Reduction of energy loss by mechanical friction has been strongly required in recent years for improving fuel efficiency especially for automotive engine. Even at a modest rate, it is of primary importance to reduce parasitic energy losses and provide environmental sustainability. An approach to achieve this target is the development of new lubrication technologies, combining new lubricant formulations and cutting-edge coatings transferable to industrial applications. Since superhard carbon material like tetrahedral amorphous carbon (ta-C) and Nano-Crystalline Diamond (NCD) coatings combine both crucial properties, high hardness with an ultra-smooth surface roughness, they have attracted a growing interest in the last decade. While the friction coefficient is generally extremely high under ultra high vacuum conditions, in the earlier years we have discovered the ability of these coatings to be lubricated by selected biodegradables green molecules like fatty acids, glycerol mono-oleate GMO and polyols, as well as their mixture with synthetic base oil such as Poly-Alpha Olefines (PAO). Some of these compounds are able to lubricate ta-C and NCD coatings with a friction coefficient below 0.01 (so-called superlubricity) in thin-film EHL/mixed regime and below 0.03 in severe boundary regime without significant wear1. As this case of superlow friction is extremely promising for many applications fields, the related mechanism has been investigated and a special attention has been paid to the surface chemistry and structural changes of the carbon coatings. By coupling advanced extreme surface analyses (PES and XANES), we show that the mechanism of friction reduction is related to the tribo-formation of quasi-2D planar graphene-like structures at the top of the colliding asperities (thickness about 1 nm). Eventually, the graphene can be slightly oxidized by the OH-groups coming from the tribo-decomposition of the lubricant molecules trapped between asperities. Moreover, the rubbed sub-surface is enriched with sp2-hybridized carbon, such as in a soft a-C material, during the friction2. These strong structural changes certainly ease the tribochemical-formation of the carbon rings present in the graphene-like structure as pointed out by atomistic computer simulations.

1. M. Kano, J. M. Martin, K. Yoshida, M.I. De Barros Bouchet, Friction J., 2 (2) (2014) 156.

2. M.I. De Barros Bouchet, J.M. Martin, J. Avila, M. Kano, K. Yoshida, T. Tsuruda, S. Bai, Y. Higuchi, N.i Ozawa, M. Kubo and M. C. Asensio, Scientific Reports, 2017 (DOI: 10.1038/srep46394).

8:40am **TR+AC+TF+VT-ThM3 Role of Deuterium and Hydrogen in the Physical Understanding of Nano-friction in a-C:H/D Thin Films**, *F.G. Echeverrigaray, S.R. Sales de Mello, A.F. Michels,* UCS, Brazil, *F. Alvarez,* UNICAMP, Brazil, ***Carlos Figueroa***, UCS, Brazil

The friction phenomenon is a complex manifestation of the nature. In spite of phenomenological laws can describe the friction force at different scales, the fundamental physical understandings of such a phenomenon do not have consensus. Phenomena such as phononic, electronic, magnetic, and also electrostatic effects and models were developed in order to explain the meta- and nano-friction behavior of materials. In this work, we report the friction behavior of a diamond spherical dome sliding on different amorphous carbon thin films containing different amounts of hydrogen and/or deuterium inspecting at the meta-nanoscale indentation. Two important situation are reported. Firstly, for samples where hydrogen was replaced by deuterium in the thin film bulk, the friction coefficient decreases for increasing *deuterium* included in the carbon underneath structure. Secondly, for samples where hydrogen content is increased on the surface, the friction coefficient decreases with the increasing of the ratio H/C at the surface. In this paper, we discuss two different physical mechanisms describing these peculiar experimental results: dissipation effects associated with phonon coupling and van der Waals forces contributions coexisting and determining the friction behaviour of a-C:H/D for the cited studied situations.

9:00am **TR+AC+TF+VT-ThM4 Imaging X-Ray Absorption Spectroscopic Investigation of the Mechanisms Behind the Environmental Dependence of the Tribological Properties of Amorphous Carbon Surfaces**, ***Filippo Mangolini***, University of Leeds, UK, *M. Koshigan,* Ecole Polytechnique Montréal, Canada, *M.H. Van Benthem, J.A. Ohlhausen,* Sandia National Laboratories, *J.B. McClimon, J. Hilbert,* University of Pennsylvania, *J. Fontaine,* Ecole Centrale de Lyon, France, *R.W. Carpick,* University of Pennsylvania

Among the variants of diamond-like carbon films developed for the ever-increasing performance and durability requirements of tribo-mechanical applications, silicon oxide-containing hydrogenated amorphous carbon (a-C:H:Si:O) is of interest as it exhibits good tribological performance across a broader range of environments compared to hydrogenated amorphous carbon, and higher thermo-oxidative stability. However, the scientific basis for this improved behavior is not established. In this work, we develop a fundamental understanding of the structural transformations occurring in a-C:H:Si:O when sliding against steel in different environments (from high vacuum to controlled hydrogen and oxygen pressures). The results of tribological experiments revealed that upon increasing the oxygen pressure in the experimental chamber from 10 mbar to 1000 mbar, the coefficient of friction increased from 0.02±0.01 to 0.06±0.01, whereas upon increasing the hydrogen pressure from 50 mbar to 2000 mbar, the coefficient of friction decreased from 0.08±0.01 to 0.02±0.01.The subsequent near edge X-ray absorption fine structure (NEXAFS) spectroscopic measurements and X-ray photoelectron spectroscopy (XPS) analyses provided insights into the structural transformations and chemical reactions occurring in a-C:H:Si:O upon sliding. Independently of the gas, a stress-induced conversion from sp^3- to sp^2-bonded (disordered) C-C bonds occurs. When sliding in hydrogen, the newly-generated, strained sp^2 carbon layer reacts with hydrogen molecules to form a hydrogenated amorphous carbon interfacial material. Upon increasing the hydrogen pressure, the fraction of C-H bonds increases in the near-surface region of the wear tracks formed on a-C:H:Si:O. This is proposed to progressively lower the shear strength of the material at the sliding interface, thus resulting in a decrease of friction with hydrogen pressure. When sliding in oxygen, the dissociative reaction of oxygen molecules with strained sp^2 C-C bonds leads to the formation of C=O groups. Additionally, increasing the oxygen pressure during tribological testing leads to an increase in oxygen concentration in the near-surface region of a-C:H:Si:O together with an increase in the fraction of Si atoms in high oxidation states. These surface chemical changes and structural transformations are proposed to increase friction with oxygen pressure by progressively increasing the shear strength of the material generated at the sliding interface.

9:20am **TR+AC+TF+VT-ThM5 Structure Evolution in Tribological Interfaces Studied by Multilayer Model Alloys**, ***Martin Dienwiebel***, *E. Cihan*, Karlsruhe Institute for Technology (KIT), Germany **INVITED**

During sliding of surfaces the near surfaces undergo significant changes in terms of topography, composition and microstructure and a so-called “third body” or “tribomaterial” forms which differs strongly from the bulk materials in terms of topography, composition and microstructure. Initially we use multilayer model alloys of an Au/Ni layer system to study effects of grain size on steady-stady friction by varying the layer spacing and the number of layers. Experiments are performed in a UHV microtribometer, the analysis of microstructure before and after tribological stressing is done by FIB and STEM and the chemistry is investigated by XPS.During sliding of surfaces the near surfaces undergo significant changes in terms of topography, composition and microstructure and a so-called “third body” or “tribomaterial” forms which differs strongly from the bulk materials in terms of topography, composition and microstructure. Initially we use multilayer model alloys of an Au/Ni layer system to study effects of grain size on steady-stady friction by varying the layer spacing and the number of layers. Experiments are performed in a UHV microtribometer, the analysis of microstructure before and after tribological stressing is done by FIB and STEM and the chemistry is investigated by XPS.

11:00am **TR+AC+TF+VT-ThM10 Carbon, Carbon Everywhere, from Catalysts to Hip Implants**, ***Laurence Marks***, Northwestern University **INVITED**

Friction is a pervasive problem, by some estimates consuming about 5% of the GDP of the economies of the developed world, and a recent analysis has indicated that about one third of the fuel energy in automobiles goes to overcoming frictional losses. While the importance of minimizing friction can be traced back at least as far as the tomb of Tehuti-Hetep, circa 1880 B.C, where a man can be seen pouring a lubricant to assist moving a statue, there are still many unknowns in the field of tribology which encompasses friction as well as other critical processes such as wear and lubrication. For many of

the phenomena in tribology there are still numerous unknowns. When it comes to biological systems most work to date has assumed that different mechanisms are taking place than occur for inorganic systems. While there are differences, there are also significant similarities particularly in implants when inorganic and biological meet. We have recently shown that for metal implants in-vivo the same type of graphitic materials that appear in many areas ranging from heterogeneous catalysis to oil lubrication are present. Beyond just lubrication, corrosion of these materials is not special to biological systems, but has strong similarities to established factors such as grain boundary precipitation and a significant role for molybdenum as reducing the occurence of breakdown of the protective oxide film via complex processes involving solute trapping. These and related recent results will be described.

11:40am **TR+AC+TF+VT-ThM12 Tribology of Cellular Interfaces**, ***Angela Pitenis***, *J.M. Urueña, S.M. Hart, T.T. Hormel, C.S. O'Bryan, S.L. Marshall, K.D. Schulze, P.P. Levings, T.E. Angelini, W.G. Sawyer*, University of Florida **INVITED**

Human health, mobility, and quality of life critically hinge on the body's ability to provide adequate lubrication between most contacting and sliding biological interfaces. Soft, aqueous, and mucinated biopolymer networks lining all moist epithelia enable the body to provide lubricity over a wide range of contact pressures and sliding speeds. The exquisite slipperiness and softness of biological sliding interfaces present significant experimental challenges for fundamental studies on their tribological performance. Physiological contact pressure conditions must be matched in in vivo, ex vivo, and in vitro studies that aim to acquire physiologically-relevant friction measurements. While biotribological investigations using living cells, cell layers, and tissues necessitate low contact pressure measurements, such studies frequently rely on the application of low forces to achieve accomodating contact pressures (kPa range), and traditional methods can decrease the contact area below a physiologically-relevant threshold. The softness of a cell layer (E ~ 10 kPa) provides an order-of-magnitude estimate for the amount of mechanical pressure that may be applied to cells during tribological testing; contact pressures about 5 kPa and shear stresses in excess of 200 Pa are sufficient to wreak significant damage to a cell layer. Recently, direct contact tribological experiments on a living cell layer without incurring any measurable cell death in the sliding path has become possible through the application of a soft, thin, spherically-capped membrane hydrogel probe. With this experimental configuration, in vitro tribological experiments were performed against a monolayer of mucin-producing human corneal epithelial cells (hTCEpi) for 10,000 reciprocating cycles at physiologically-relevant contact pressures and challenging sliding speeds. The gel-cell sliding interface under applied normal loads of ~ 200 μN resulted in measured friction coefficients of $\mu \sim 0.06$ and achieved shear stresses on the order of 60 Pa, which is below the critical shear stress for inducing cell death; excellent cell survival rates (~99.8%) were measured after extended duration tribological experimentation.

Plasma Science and Technology Division
Room: 22 - Session PS+VT-ThA

Plasma Diagnostics, Sensors and Control

Moderator: Aranka Derzsi, Wigner Research Centre for Physics, Hungarian Academy of Sciences, Hungary

2:20pm **PS+VT-ThA1 Quantitative Analysis of Composition and Temperature of Semiconductor Processing Plasmas via Terahertz Spectroscopy**, ***Yaser Helal***, *C.F. Neese, F.C. De Lucia*, The Ohio State University, *A. Niabati, M. Johnson, B. Craver, P.J. Stout, M.D. Armacost*, Applied Materials, Inc.

Processing plasmas are at a similar pressure and temperature to the environment used to study atmospheric and astrophysical species in the terahertz (THz) spectral region. Many of the molecular neutrals, radicals, and ions present in processing plasmas have been studied in the laboratory and their absorption spectra have been cataloged or are in the literature for the purpose of astrophysical study. Recent developments in THz devices have made technology commercially available for applications outside of specialized laboratories. The methods developed over several decades in the THz spectral region for these laboratory studies are directly applicable to diagnostic measurements in the semiconductor manufacturing industry. In this work, a continuous wave, intensity calibrated THz absorption spectrometer was developed as a remote sensor of gas and plasma species. A major advantage of intensity calibrated rotational absorption spectroscopy is its ability to determine absolute concentrations and temperatures of molecular species from first principles without altering the plasma environment. An important part of this work was the design of the optical components which couple 500 – 750 GHz radiation through a commercial inductively coupled plasma (ICP) chamber. The measurement of transmission spectra was simultaneously fit for background and absorption signal. The measured absorption signal was used to calculate absolute densities and temperatures of polar species. Examples of measurements made in ICPs will be presented. Also, time resolved measurements were made and the time evolution of molecular densities will be discussed.

2:40pm **PS+VT-ThA2 *In Situ* Measurement of Electron Emission Yields from Plasma-Exposed Surfaces**, ***Mark Sobolewski***, National Institute of Standards and Technology

Surfaces exposed to plasmas are bombarded by energetic particles which may induce electron emission. The emitted electrons may in turn influence the plasma. Accurate plasma simulations require knowledge of the flux or yields of emitted electrons. Yields can be measured directly in beam studies, but it is impractical to produce a beam of each possible energetic particle that could be produced by typical plasmas. In contrast, in-situ measurements, performed during plasma exposure, may provide useful values or bounds for effective or total electron emission yields, summed over all (or some subset) of the energetic particles present for given plasma conditions. Here, measurements were performed at 10 mTorr (1.3 Pa) in an inductively coupled plasma system equipped with an electrostatic shield and variable-frequency rf substrate bias. An insulating cap is placed on the rf-biased electrode to minimize edge effects. The cap also reduces the effective electrode size, which further limits any undesired effects of rf bias on the plasma and allows yield measurements on small substrates. The rf voltage and current across the sheath adjacent to the rf-biased electrode are measured and analyzed by detailed, numerical sheath models, which allow the current of electrons emitted from the surface to be distinguished from other mechanisms of current flow. The observed dependence on voltage and rf phase allows some discrimination between emission induced by energetic positive ions and that induced by photons and metastables. The technique is validated by comparing measurements made in argon discharges with literature results from beam studies and then is applied to plasma etching discharges in fluorocarbon gas mixtures.

3:00pm **PS+VT-ThA3 Studying Dynamic and Structured Plasma Systems Utilizing Laser-Collision Induced Fluorescence**, ***Edward Barnat***, *A. Fierro*, Sandia National Laboratories **INVITED**

Laser collision-induced fluorescence (LCIF) is a powerful diagnostic which can be used for making temporally and spatially resolved measurements of electron densities in a plasma discharge. The technique, which involves the measurement of optical emission emanating from higher energy excited states due to the redistribution of the lower energy laser-excited state by collisions with energetic plasma species, has been readily employed to study both helium and argon discharges. In this presentation, an overview of the fundamental principles and anticipated limitations of the LCIF method will be presented. Examples of the LCIF method applied to structured and dynamic discharges generated in helium and argon will be presented to demonstrate the utility of this diagnostic technique. Finally, recent efforts used to extend the LCIF method to higher pressure (near atmospheric pressure) discharges will be discussed.

4:00pm **PS+VT-ThA6 Effect of Ion Inertia on Ion Energy Broadness on Biased Electrode in Dual Frequency Capacitively Coupled Argon Plasma**, ***Yunchang Jang***, *H.-J. Roh, N.-K. Kim, S. Ryu, G.-H. Kim*, Seoul National University, Republic of Korea

Ion response time to RF sheath voltage is important to control the energy spread of ion energy distribution (IED) in the dual frequency capacitively coupled argon plasma. IED is known as being governed by the dynamics of ion in RF sheath and the magnitude of RF voltage peak. In previous study, semi-analytic models to determine IED were derived from concept of ion response time (τ_i). Ion energy broadness (ΔE_i) was represented in terms of the sheath voltage oscillation (V_{pp}) and τ_i/τ_{rf}. Ion response time was assumed as ion transit time across the sheath, τ_{ion} by adopting correction factor without thorough understanding. In this study, we investigate the underlying physics of the correction factor, consequently defining the ion response time τ_i with RF sheath voltage oscillation. Experiment were performed in dual frequency CCP at 20 mTorr of argon gas which has the ratio of maximum sheath size to ion mean free path ~ 2. Various ranges of RF bias (from $\tau_{ion}/\tau_{rf} \sim 0.05$ to $\tau_{ion}/\tau_{rf} \sim 10$) were applied to bottom electrode to enhance the incident ion energy with very high frequency (VHF, $\tau_i/\tau_{rf} \sim 10$) applied on the top electrode (showerhead) to sustain plasma. A commercial retarding field analyzer (Impedans, Vertex V4.0.10) was employed to measure IED. Plasma density, electron temperature and plasma potential were measured by using RF compensated Langmuir probe. Experimental results of ΔE_i to V_{pp} were compared with models under assumptions that ion response time is ion transit time across the sheath (τ_{ion}) or one of ion plasma frequency ($1/\omega_{pi}$). Experiment results revealed that the time scale of ion response time is determined by $1/\omega_{pi}$ rather than τ_{ion} in this high-density plasma. This result implies that ion response time is governed by the ion inertia at the sheath boundary to RF sheath oscillation. Ion inertia becomes the initial condition of ion acceleration and govern the ion energy arriving at surfaces.

4:20pm **PS+VT-ThA7 Collision Frequency Estimation using Microwave Hairpin Resonator Probes**, *D. Peterson*, ***Steven Shannon***, North Carolina State University

Microwave hairpin resonator probes have become convenient alternatives to Langmuir probes to measure electron density in low temperature plasmas. The impact of electron collisions with neutrals with regard to the analysis of the resonant frequency shift from which this density is determined has been well established.[1] In this work, a method for extracting the electron neutral collision frequency by measuring resonance broadening due to collisions is presented. By using both the resonance frequency and collision-broadened resonance width, the electron density and electron neutral collision frequency can be measured. Measurements are made in argon, oxygen, Ar/O_2, and helium plasmas sustained in an RF driven capacitive coupled parallel plate system operating in the 100's of mTorr to Torr range. Comparisons to calculated and modeled collision frequencies in single component background gases (Ar and He) are made; experimental results agree well with these conditions. Collision frequency measurements in more challenging regimes including molecular gases and gas mixes are also presented to demonstrate applicability across a broad range of pressures and gasses. Probe design, analysis methodology, and parametric trends in capacitive systems with regard to gas density, electron density, power density, and gas composition will be presented. This work is supported through a generous gift by Applied Materials Inc.

[1] *Plasma Sources Science and Technology* 16, no. 4 (2007): 716

4:40pm **PS+VT-ThA8 In-Situ Diagnostics of Processing Plasma and Semiconductor Films for High-Efficiency Silicon Hetero-Junction Solar Cells**, ***Shota Nunomura***, National Institute of Advanced Industrial Science and Technology (AIST), Japan **INVITED**

The plasma processing is a key technology for fabricating semiconductor devices such as solar cells, light-emitting diodes and transistors. In those devices, the semiconductor films are often prepared and/or post-processed by various plasma processes. During the processes, the films are exposed into the UV, radicals, and ions, and thereby the electronic property of the films is often degraded. So, the investigation of the plasma-material interaction is important for understanding the degradation mechanism and also for further developing the plasma processing technology.

Here, we show in-situ characterization of the electronic property of semiconductor films as well as the gas-phase plasma diagnostics during the plasma process. The process we diagnosed was PECVD of hydrogenated

amorphous silicon (a-Si:H) for the passivation of silicon heterojunction solar cells. The plasma parameters and gas-phase spices, such as ions, radicals, and precursors are measured by using conventional techniques of Langmuir probe, quadrupole mass spectroscopy and optical emission spectroscopy [1-3]. Together with this gas-phase diagnostics, we measured the transport properties of the growing a-Si:H films such as carrier transport and trapping, by using a recently developed optical pump probe technique [4,5]. The optical property such as the bandgap, refractive index and extinction coefficient, was also characterized by real-time spectroscopic ellipsometry.

We found that transport property of the a-Si:H films was strongly limited by the defects generated during the PECVD process, and improved by post-deposition annealing process. The generated defects were distributed near the film surface; the defect rich surface layer was estimated to be less than approximately 10 nm. As for defect annihilation, the post deposition annealing was very efficient. The annealing temperate and period strongly influence the defect relaxation, inducing the improved carrier transport. The relation between the plasma process and transport property will be described in the presentation.

[1] S. Nunomura, I. Yoshida, and M. Kondo, *Appl. Phys. Lett*. **94**, 071502 (2009). [2] S. Nunomura and M. Kondo, *J. Appl. Phys.* **102**, 093306 (2007). [3] S. Nunomura. H. Katayama I. Yoshida, *Plasma Sources Sci. Technol*. **26**, 055018 (2017). [4] S. Nunomura, I. Sakata, and M. Kondo, *Appl. Phys. Express* **6**, 126201 (2013). [5] S. Nunomura and I. Sakata, *AIP Advances* **4**, 097110 (2014).

5:20pm **PS+VT-ThA10 Towards *In Situ* Microwave Imaging in Plasmas**, *A. Tselev*, University of Aveiro, Portugal, *J. Fagan*, NIST, ***Andrei Kolmakov***, CNST/NIST

There exists a great need for *in situ* nanoscale characterization of surface/interface morphologies during plasma treatments. These include plasma induced growth, surface modification, sputtering and other processes relevant to semiconductor and aerospace industries, environmental remediation and biomedical applications. To address these needs, the current approaches rely on either "post mortem" sample microscopy or *in situ* optical analytical methods. The latter, however, lack required nanoscale spatial resolution.

In this communication, we propose to use near-field microwave imaging known as scanning Microwave Impedance Microscopy (sMIM) to image processes in plasma. Different to optical microscopy, the sMIM is sensitive to variations of local permittivity and conductivity of the material under a scanning probe. We demonstrate applicability of the sMIM to monitor plasma-assisted processes with a submicron spatial resolution. In our approach, a plasma environment with an object of interest is separated from the sMIM probe and the rest of the microscope by a SiN membrane of a few-10s nm thickness, and the imaging is performed through this membrane. As a proof of concept, we were able to image carbon nanotube films drop-casted onto the SiN membranes and their transformations in the process of plasma-induced oxidation by a low-pressure air plasma. To the best of our knowledge this is the first report on application of an SPM for in situ imaging of plasma processing. The experiential limitations such as electromechanical and thermal stability of the membranes will be discussed.

5:40pm **PS+VT-ThA11 Probe System for Radical Species Characterization in Vacuum with Centimeter Spatial Resolution**, ***Ivan Shchelkanov***, *D. Qerimi, A. Hayes, J.T. Wegner, D.N. Ruzic*, University of Illinois at Urbana-Champaign

Among plasma diagnostics one of the most difficult tasks is getting an estimate of radical gas species concentration in the ground state without plasma presence in the diagnosed volume. This is probably the major task for characterisation of downstream plasma composition in various areas of applied plasma technology and the requirements for characterisation tool are very strict. The ultimate device should have a good spatial resolution, relatively high response time, operate in highly reactive plasmas and in presence of sputtering flux materials, should be capable to characterize species of unknown geometrical distribution and composition.

The idea of a tool, which could satisfy most of the mentioned requirements, was proposed more than ten years ago [1] but only recently the Center for Plasma Materials Interaction was able to develop a complex system which can measure composition and density of radical species with 1 cm spatial-resolution and response time of 15 seconds in the presence of high intensity RF fields and flux of sputtered material. The system can measure density of oxygen, nitrogen, and hydrogen radicals, when different species present in the chamber at the same time. For vacuum chamber of 13 inch in diameter and 46 inches tall, which is equipped with 1 kW Helicon plasma source , the measured density at ~75 mTorr, 1kW power and 10 inch from the source, the density of radical species of hydrogen was 0.7 [±0.5] $*10^{21}$ m^{-3} and of nitrogen radicals it was 1.1 [±0.7] $*10^{20}$ m^{-3}. Additional comparison with zero dimensional model showed a match with-in an errorbar between an experiment and the model.[2]

The principle of the radical species concentration measurement is the following. The thermocouple tip is coated with a particular catalytic metal. Once the probe is exposed to the gas atoms, recombination of gas atoms occurs on the surface of the probe tip. The catalytic surface provides efficient recombination thus more energy is delivered to the surface from the recombination reaction [3] compared to a probe tip without the catalytic surface. By measuring the temperature of the probe it becomes possible to quantify the amount of gas atoms in the probe vicinity. Different radical species can be distinguished by using catalytic surfaces particular to the species in question. Current work is focused on radical probe system capabilities, physical limitations, and examples of characterized plasmas.

References:

1. M. Mozetic / Vacuum V.47 #6-8 pages 943 to 945 (1996)

2. D.T. Elg / *J. Micro/Nanolith. MEMS MOEMS, 16, 023501 (2017)*

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6:00pm **PS+VT-ThA12 Spatiotemporal Evolution of RF Magnetic Field and Plasma Current in a Very High Frequency Plasma Source**, ***Jianping Zhao***, *P.L.G. Ventzek, B. Lane, C. Campbell*, Tokyo Electron America, *T. Iwao, K. Ishibashi*, Tokyo Electron Limited

Large-area plasma processing systems capacitively driven at very high frequencies (VHF, e.g. 100MHz) have attracted much interest for semiconductor device and flat panel display processing. VHF has the advantage of generating plasma with more efficiency as power is coupled more into electrons and less into ions in the sheath. Benefits are seen for processes requiring reduced ion bombardment energy, ostensibly to minimize damage, high ion and radical flux to the substrate. The benefits of VHF are accompanied by challenges. The short wavelength associated with VHF source power is reduced even further in the presence of high density plasma. The wavelengths are comparable to the RF electrode dimension. High plasma densities can also lead to skin effects that screen the electromagnetic fields from parts of the plasma. As a result, spatial variations in plasma density and sheath voltage can arise and lead to undesired non-uniformities in process parameters such as etch or deposition rate. E to H transitions and plasma-sheath local resonances are other potential destroyers of plasma uniformity. Resonances and mode jumping can potentially prevent smooth plasma property control through adjustable process parameter changes. In order to understand these fundamental electromagnetic effects on VHF plasma non-uniformity to achieve a better design of plasma source, it is desired to have a detailed investigation on the spatial and temporal evolution of RF magnetic field and plasma current spanning a large RF power, pressure, and chemistry range. We present here a time and phase resolved measurements of the spatial structure of the electromagnetic waves in a 100MHz plasma source performed with a magnetic field probe (B-dot loop). The probe was translated across the diameter of the VHF plasma, measuring the magnitude and phase of the fundamental and harmonics of the plasma excitation frequency as a function of radial position. The measured magnetic fields displayed a transition from simple to complex behaviors depending on plasma conditions. The spatiotemporal resolved magnetic field exhibits a series of fast current reversal and subsequent circulation driven by inward wave propagation that are electromagnetic in nature. We show how the onset, frequency and amplitude of the current reversal and subsequent circulation are strongly related to applied plasma conditions (e.g., density, sheath thickness). We also show that plasma current derived from the magnetic field distribution is closely correlated to the plasma density profile measured by a plasma absorption probe.

Vacuum Technology Division
Room: 9 - Session VT-ThA

Surface Science for Accelerators

Moderators: Jay Hendricks, NIST, Alan Van Drie, Tri Alpha Energy, Inc.

2:20pm **VT-ThA1 Adsorption/Desorption from Amorphous Carbon Coating at Cryogenic Temperatures**, ***Anne-Laure Lamure***, *V. Baglin, P. Chiggiato, B. Henrist*, CERN, Switzerland **INVITED**

The CERN Large Hadron Collider (LHC) is the world's biggest particle storage ring. Particles circulate in a 27km pipe, under vacuum. One of the main vacuum limitations is the electron cloud.

Photoelectrons are produced when the synchrotron radiation from the proton beam hits the wall. They are then accelerated toward the beam, gain energy and extract new electrons by secondary electron emission. The avalanche phenomenon which is observed is called multipacting.

Electron cloud is deleterious as it interacts with the beam, induces gas desorption and produces additional heat load on the cryogenic system of the magnets.

In order to mitigate the multipacting effect for the upgraded LHC (HL-LHC), amorphous carbon, with a low secondary electron yield, will be coated in some cryogenic magnets.

In this context, it is important to know the behaviour of the usual residual gas (H2, CO, CH4, CO2) on amorphous carbon coating held at cryogenic temperature, in order to know how to operate the vacuum in the accelerator. The quantity of gas that can be stored on the surfaces and the binding energy of adsorption are two highly interesting information.

The results of two types of experiments will be presented. Adsorption isotherms give the vapor pressure depending on the coverage of gas on the surface. Isotherms of H_2 at 4.2K and of CO and CH_4 at 77K have been measured.

Thermal Desorption Spectroscopy, that allow to determine the average binding energy between the gas and the surface, have been carried out for the four gases, for different initial coverages.

It has been measured that amorphous carbon is a porous material which can store more gas at cryogenic temperature than usual technical surfaces such as copper or stainless steel. The consequences for the accelerator will be discussed. A model to compute the pressure rise in the vacuum pipe, depending on the temperature variation and on the initial coverage, is under development.

3:00pm **VT-ThA3 Heavy ion-induced Desorption and its Impact on Next Generation Accelerators**, ***Markus Bender***, *H. Kollmus*, GSI Helmholtzzentrum für Schwerionenforschung GmbH, Germany, *E. Mahner*, CERN, Switzerland **INVITED**

Dynamic pressure increases in vacuum systems of particle accelerators have been observed since almost 50 years. Since the turn of the millennium, the dynamic vacuum turned out to be an intensity limitation in particle accelerators, e.g. in the Low Energy Antiproton Ring (LEAR) at CERN or the heavy ion synchrotron SIS18 at GSI. Here, charge exchanged lost beam ions stimulate the release of gas from the chamber walls and the subsequent pressure increase leads to increased beam-loss. Hence the effect is self-amplifying and can lead to severe deterioration of the vacuum to the point of complete beam-loss. Consequently heavy ion-induced desorption is an issue for next-generation heavy ion accelerators such as the FAIR facility or Spiral2 with highest beam intensities.

To come up against this dynamic vacuum effect, several measures have been conducted. In particular the physics behind the ion-induced release of gas was investigated. For that purpose, several samples have been irradiated with ion beams of different parameters and the resulting desorption yields have been measured. A broad range from some 10 to several 10,000 released gas molecules per incident ion was observed. From the gathered results a clear picture of the underlying process of ion-induced desorption was drawn. It could be shown that the desorbed gas is originating mainly from the surface or surface-close regions of the target. But in contrast to earlier ideas, sputtering of the oxide layer on metals was not identified as the source for the desorbed gas. Latest experiments prove that pre-treatment of critical components is most important to minimize the desorption yield and therefore, especially thermal annealing was investigated in detail.

Besides experimental findings a model calculation was developed that is able to describe and compare desorption yields of different collision systems. The calculation is based on the inelastic thermal spike model and describes ion-induced desorption as enhanced thermal desorption due to a transient overheated spot around the ion impact.

Presently we are able to propose materials, coatings, and treatment procedures for best performance in particle accelerator vacuum systems.

4:00pm **VT-ThA6 Outgassing Behavior of Different Oxide Ceramic Materials**, ***Katharina Battes***, *C. Day*, *V. Hauer*, Karlsruhe Institute of Technology (KIT), Germany

In general, ceramics show interesting mechanical, thermal and electrical properties and are supposed to have relatively low outgassing rates. Therefore, in vacuum applications they are often used for feedthroughs for example. However, quantitative numbers on outgassing of most of the ceramic materials are hard to find in literature.

For this reason the outgassing of different ceramic materials was studied at the Outgassing Measurement Apparatus (OMA), which uses the difference method. First, oxide ceramics like alumina, magnesia, silica, and MACOR®, which consists of silica and other oxide ceramics, were measured. All measurements were performed at room temperature, 100 °C and 200 °C to investigate the temperature behavior of outgassing. Additionally, the outgassing species were determined by a quadrupole mass spectrometer.

The paper shows quite low outgassing rates for most of the examined ceramics. After 100 h at room temperature an outgassing rate of about $2 \cdot 10^{-8}$ $(Pa \cdot m^3)/(s \cdot m^2)$ is achieved for alumina for example. The mass spectra show similar residual gas spectra as seen for metals. Thus, these materials can be used in ultra-high vacuum applications.

4:40pm **VT-ThA8 APS-Upgrade Storage Ring Vacuum System Sector Mockup and Vacuum R&D Activities**, ***Jason Carter***, Argonne National Laboratory

As the APS Upgrade project continues in its preliminary design phase the APS-U storage ring vacuum system plans continue to mature while ongoing R&D activities and analysis are validating and strengthening the design. The storage ring magnets and structural support designs constrain the system to have narrow aperture vacuum chamber dimensions and limit allowable UHV pumping elements and locations. Monte-Carlo vacuum system analysis has indicated that the pressures and performance should meet requirements during and after accelerator commissioning. The margin of error for analysis must be better understood so a number of ongoing R&D efforts are helping to better predict and improve the performance.

A 28 meter length full sector vacuum system mockup will be installed in Fall 2017 and will include prototype vacuum chambers and all pumping elements. A sector mockup vacuum test plan will be presented which examines pumping speeds, outgassing rates, and the pumping conductance. NEG coating performance will be key to APS-U vacuum success and prototypes and further analysis are helping evaluate current coating plans and the option of adding more coatings. Finally, R&D proposals are progressing to measure photon stimulated desorption from APS-U style chamber designs.

5:00pm **VT-ThA9 Numerical Tools for Particle Accelerator Vacuum Systems**, ***Giulia Lanza***, SLAC National Accelerator Laboratory

A number of different gas density simulation programs have been applied in the design of the SLAC National Accelerator Laboratory's LCLS-II accelerator's vacuum system. Starting from these basic programs, this talk gives an overview of the available numerical methods for the analysis and design of a linear accelerator vacuum system.

Programs like Pressure5, LTspice, Vaccalc, Molflow+ and others are described and compared. Their optimal domain of applicability, the pros and cons are discussed.

5:20pm **VT-ThA10 Developing Particle Control Infrastructure for the ESS High Beta Project at STFC Daresbury Laboratory**, ***Mark Pendleton***, STFC Daresbury Laboratory, UK

As part of a UK In-Kind contribution to the European Spallation Source (ESS), STFC-Daresbury laboratory has agreed to procure, fabricate, test and deliver 84 + 4(5-cell) high-beta superconducting 704.41 MHz dressed cavities according to ESS requirements.

As part of the test phase and re-work at STFC the cavities will have to be processed and connected in a particulate controlled environment.

This paper will describe the developments of the Main Cleanroom facility which will be utilised for the workflow of the High Beta Cavity to undertake a High Pressure rinse cycle. It will also cover the Cryostat Insert stands and the design of the Modular cleanroom solutions that will be utilised to connect the cavity under ISO 4 conditions to the new Vertical Test Cryostat Insert developed at STFC.

5:40pm **VT-ThA11 Functional Coatings for Gauges and Components**, *B. Andreaus*, *C. Strietzel*, ***Martin Wüest***, INFICON Ltd., Liechtenstein, *C. Guerra-Nuñez*, *M. Ruoho*, *I. Utke*, *J. Michler*, *X. Mäder*, *M. Polyakov*, Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

Process industry is constantly changing. New manufacturing processes using new chemistries are developed. Yet, quality and cost pressure demand that processes are highly reliable, repeatable and need fewer maintenance interruptions. For vacuum sensors this means that they need to have a longer life in process before they need to be exchanged due to sensor degradation caused by process corrosion. To cope with this, we have investigated ways to protect the vacuum sensors from process related influences. Coatings are a good way to protect a surface from corrosion while leaving the underlying structural part without change. Coatings can be adapted to the changing customer needs. We will present results from experiments we have done with gauges and components using different protective layers.

6:00pm **VT-ThA12 60 Years of Ion Pumps: From the Invention to the Latest Developments**, ***Mauro Audi***, Agilent Technologies, Italy

Since their invention in 1957 at Varian Associates as a pumping device for electron tubes at relatively high pressures , Ion Pumps have continuously moved towards lower pressures , and nowadays they are the pumps of choice for most of UHV applications in both research and industrial field

This includes a large a variety of high and ultra high vacuum systems , from Particle Accelerators to Synchrotron Light Sources and Gravitational Wave Detectors , from Scanning Electron Microscope to Surface Analysis and Medical Equipment

Application requirements have changed dramatically in these 60 years in terms of starting and operating pressures , pumping performances , ability of pressure reading , cleanliness , particle emissions , safety , resistance to radiation .

The latest developments on Ion Pump Technology are presented , including :

- a new combination of magnetic field and cell dimensions to realize the first ion pump with the maximum pumping speed in the low pressure range

- a new vacuum firing process to minimize the outgassing and reduce particles

- an anode design that minimizes the field emission and the leakage current , and additional shields that minimize charged particle emissions

-a controller design that allows starting Ion Pumps with a very limited power and can vary voltage supplied to the ion pump in order to optimize both the pumping performances and the pressure reading ,

- a combination of ion pumps with NEG pumps in order to reach the lowest ultimate pressure

Authors Index

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