

Vacuum Technology

Room: 14 - Session VT-MoM

Vacuum Gauging and Metrology

Moderator: G.A. Brucker, Brooks Automation, Inc.,
Granville-Phillips Products

8:20am **VT-MoM1 Sapphire-based Capacitance Diaphragm Vacuum Gauge Operating at 500 °C.** *T. Ishihara, H. Tochigi, J. Yoshinaga, M. Nagata*, Azbil Corporation, Japan

Growing demand of low temperature processing for environmental concern in semiconductors and related technologies, such as organic electroluminescent display processing, now require process materials handling temperature to be higher (250-500°C) than it used to be, especially in vacuum deposition processing. In these applications, self-heating absolute manometers consist of nickel base alloy or ceramics operated up to 200°C are used to avoid deposition. Because of the plasticity of the diaphragm itself or bonding materials, if self-heating temperature becomes higher, characteristics of sensors become worse. So there is strong demand for manometers, which operate stable at 250-500°C. Authors have developed entirely sapphire-based capacitive pressure sensor chip utilizing Micro-Electro-Mechanical Systems technologies that is suitable for high temperature application. In this paper, we present packaging techniques of the sensor chip to construct the sapphire-based capacitance diaphragm vacuum gauge. Fig.1 and 4 show schematic cross-sectional views of a sapphire CDVG, bonding interface, and sensor chip respectively. In the pressure gauge with 0-133.32Pa absolute, packaging requires low mechanical stresses from the exterior metal body to the sensor chip. Generally, braze, solder, and glass are used for packaging, especially for bonding the sensor chip onto the metal body. But these intermediate bonding materials generate higher stress on sensor chip and its creep yields sensor zero point drift or span drift. To avoid these mechanical stresses, the sapphire chip is first bonded to a sapphire disc, which in turn is bonded to the metal body without any intermediate materials. We adopted solid-state bonding techniques, in which 1-10 MPa pressure is applied at bonded parts at a temperature of at least 900°C. Fig. 2 and 3 show the TEM images of bonded sapphire-metal and sapphire-sapphire interfaces used in this package respectively. These images show no observable interlayer, indicating perfect bonding at the atomic level. Fig. 5 shows the pressure sensitivities of this sensor at 500°C, in which error of the span is under 0.05Pa. In Fig. 6 the temperature dependence of zero and span of the sensor at from 200 to 500°C are presented. The span shift at this temperature range is only 0.52Pa. Fig. 7 shows the zero drift at 500°C. About 0.1%FS zero-shift can be seen for 600 hours. Sensor output resolution can be estimated at 0.0039Pa from Fig.8. These results show excellent performance of the sapphire CDVG over 200°C. With advanced packaging technologies, we have realized a sapphire-based manometer that can operate at 500°C, which enables measurement and controls of advanced processes.

8:40am **VT-MoM2 On the Stability of Capacitance Diaphragm Gauges.** *M. Wüest, C. Strietzel*, INFICON Ltd, Liechtenstein

Capacitance Diaphragm Gauges (CDG) are extensively used in critical coating industries due to their stated accuracies. However, the process to which the gauge is exposed to as well as the ambient environment can influence the accuracy of the gauge. Observed effects include e.g. zero pressure drift, span drift, noise, or depositions on the membrane. In this talk we will explore these influences on the accuracy.

9:00am **VT-MoM3 A Truly Cold Vacuum Gauge for Ultra-high Vacuum and Extreme-high Vacuum Employing a Hydrogen Absorber.** *G.A. Mulhollan*, Saxet Surface Science **INVITED**

Conventional gauging techniques for ultra-high vacuum (UHV) and extreme-high vacuum (XHV) employ a hot filament or a plasma discharge, thereby ionizing the background gas molecules so that they can be collected and signal processed, to meter the vacuum pressure. Filaments emit heat and electrons, both of which can raise the system pressure. Stray electrons and ions from cold cathode discharges can have much the same effect. XHV metering limiting characteristics include x-ray induced errors and extinguished discharges. At UHV and XHV pressures, the predominant gas species is hydrogen. Little error is incurred in the total vacuum pressure value if only the hydrogen pressure is metered via absorption. While several techniques sensitive to hydrogen adsorption in the pressure ranges of interest could be employed to take advantage of this fact, careful consideration led to the selection of the titania nanotube array as the active

element for a hydrogen absorbing vacuum gauge. Such arrays exhibit very large responses to hydrogen at atmospheric pressures.

In this relative gauging method, the titania nanotube array is mounted onto a UHV/XHV compatible header which in turn is affixed to a floating feedthrough. The feedthrough permits a bias to be applied across the array, resulting in current flow. The ensuing current flow, together with the bias value, allows an effective resistance to be calculated. The value of this resistance is proportional to the cumulative hydrogen impingement and restorative exposure history of the array, thereby enabling hydrogen as a vacuum constituent to be monitored. The metering activity is completely quiescent with respect to stray charged particle and heat generation. The ensuing gauging process has been shown to deliver excellent hydrogen gas response in vacuum. Enhanced sensitivity for XHV vacuum monitoring is achieved through illumination boosting. Alternate sensor materials, restorative methods and ultimate sensitivity limits will be discussed.

9:40am **VT-MoM5 Investigation of Pumping Combinations to Achieve XHV.** *M.L. Stutzman, P.A. Adderley*, Thomas Jefferson National Accelerator Facility, *M.A. Mamun*, Old Dominion University, *M. Poelker*, Thomas Jefferson National Accelerator Facility

The spin-polarized electron beam used at Jefferson Lab's nuclear physics accelerator is generated through photoemission from a strained-superlattice GaAs/GaAsP photocathode. The operational lifetime of the photogun depends strongly on the pressure in the system since residual gases are ionized by the electron beam and accelerated into the photocathode causing damage. To date, photoguns at Jefferson Lab have relied on NEG and ion pumps. Incremental vacuum improvements have been made through a combination of better bake protocol, chamber heat treatment, NEG pump geometry and activation protocol, improved ion pump technology and the addition of a load-lock to enable photocathode replacement without venting the photogun. This work describes pressure measurements inside a gun-style vacuum chamber with a commercial bakeable cryopump in addition to the NEG and ion pumps. Much of the talk will focus on an assessment of our ability to measure the pressure in the system using three commercial UHV/XHV vacuum gauges.

10:00am **VT-MoM6 Beyond Mercury Manometers: Are Optically-Based Primary Standards for Realization of the Pascal Within Reach?** *J.H. Hendricks, J.A. Stone, G.F. Strouse, D.A. Olson, J.E. Ricker*, National Institute of Standards and Technology

We propose to fundamentally change the method for realizing and disseminating the SI unit of pressure and vacuum, the pascal. The underlying metrology behind this advance is the ultra-accurate determination of the refractive index of gases by picometer optical interferometry. For the pascal an optical-based primary pressure standard will improve accuracy and allow the complete replacement of all mercury-based pressure standards. Pressure and vacuum standards based on refractive index could significantly reduce measurement uncertainties with the added advantage of eliminating the need for mercury manometers, which are expensive to operate and have environmental and health hazards. Mercury manometers currently serve as primary standards at 11 National Metrology Institutes (including NIST and PTB). Developing an optical-based pressure standard is central to the NIST measurement science mission, as it will improve NIST realization and dissemination of an important SI unit and will provide a needed improvement in accuracy (3X to 10X) across many industrial sectors (e.g. aerospace, energy, and advanced manufacturing) and benefit other government agencies (e.g. DoD, FAA, NASA, DARPA, and the EPA). The primary goal of the research is to develop a laser-based, SI-traceable pressure standard (1.5 ppm, k=1) along with a transportable version that can be deployed to industry covering a range of 1 Pa (vacuum) to 400 kPa (4 atmospheres pressure). The two instruments that will be developed are referred to as a variable-length optical cavity (VLOC) and a fixed-length optical cavity (FLOC). When either instrument is used as a pressure standard it is referred to as an optical interferometer manometer (OIM). The primary technical challenge involves building an apparatus to generate and precisely measure equal displacements of Fabry-Perot interferometer mirrors in vacuum and in a helium environment to picometer accuracy, thus determining refractive index in a manner that allows absolute measurement of pressure or temperature if one of these two quantities is known. More specifically, the refractive index, n , as measured by the VLOC, is intrinsically related to density such that $n-1$ depends on P/T , where P is the pressure and T is the temperature. The current state of primary mercury manometers in use at NIST will be discussed along with technical challenges of developing an optical based primary standard.

10:40am **VT-MoM8 Reduction of Statistical Scatter of Spinning Rotor Gauge Readings by Operation at Higher Rotational Frequency**, *J. Setina*, Institute of Metals and Technology, Slovenia

First spinning rotor gauge (SRG) controllers had a fixed window of operational frequency from 405 to 415 Hz. Newer controllers have a much wider range of possible rotor frequencies from 405 Hz to 810 Hz. Most users still prefer lower rotor frequencies because the SRG residual drag and its frequency dependence increases significantly with increased rotor speed. However, by increasing rotor speed the number of revolutions in a fixed sampling interval is also increased, which means reduction in statistical scatter of readings. From operational theory of SRG the standard deviation of readings shall decrease proportional to (frequency)^{-1/2} but we have found that in reality it decreases much more. At fixed sampling interval we get 4 times or more reduction of standard deviation when rotor frequency is increased from 410 Hz to 800 Hz. The reason is that when operational frequency increases, the amplitude of induced signal in pickup coils for detection of rotor frequency also increases. In a separate experiment we have observed that with stronger pickup signal the standard deviation of readings decreases. For applications where shorter response time of SRG is critical and frequency dependence of residual drag can be corrected or tolerated, we recommend operation at highest rotational frequency. We have found that typical standard deviation at rotor frequency 800 Hz and sampling interval of 5 s is smaller than at rotor frequency 410 Hz and sampling interval of 10 s.

11:00am **VT-MoM9 Long-Term Stability of Hot-Filament Metal-Envelope Enclosed Ionization Gauges**, *J.A. Fedchak*, National Institute of Standards and Technology

Hot-filament ionization gauges are used as secondary standards by calibration laboratories and as transfer standards in intercomparisons among metrology laboratories. A quantitative measurement of gauge stability with respect to the gauge calibration factor is critical for these applications. In addition, gauge stability is important for those who use gauges for process monitoring and control, and for monitoring vacuum quality. We determined the long-term calibration stability of hot-filament metal-envelope enclosed ionization gauges based upon the analysis of repeat calibrations of nine gauges over a 15 year period. All of the gauges were Bayard-Alpert type ionization gauges with an integral metal-envelope surrounding the hot-filament, grid, and collector. The gauges were calibrated repeatedly at the National Institute of Standards and Technology (NIST), but are owned by organizations external to NIST. In all cases, the gauges were removed from the NIST high-vacuum standard after calibration, shipped back to the gauge-owner, and were returned to NIST at a later date (more than 1 year) for re-calibration. Here we present results of the stability study along with discussion of the NIST high vacuum standard and ionization gauge calibration methods.

11:20am **VT-MoM10 Non-Destructive Gas Pressure Measurements Inside Sealed Vacuum Devices**, *R.S. Goeke, T.P. Hughes*, Sandia National Laboratories

Measuring the pressure inside sealed vacuum devices is a difficult proposition that typically requires destructive analysis. While commercial vacuum pressure gauges can be applied to large vacuum envelopes, the gauge's large volume and mass make them impractical for the small volumes of many vacuum devices. Incorporation of smaller volume pressure gauges such as the spinning rotor gauge or capacitance manometers can interfere with the device functionality. Measurements on these small vacuum devices can be done by a destructive technique where the device is punctured inside a calibrated ultra-high vacuum (UHV) vessel. With this technique the tube pressure is calculated from the changes in pressure of the UHV chamber and ratio of the chamber volume to tube volume. We have developed a non-destructive method by which the pressure inside high vacuum devices can be characterized without modifying or damaging the vacuum envelope. The approach transforms the existing vacuum device into a Penning or Redhead style ion gauge. We take advantage of the device features such as existing electrodes and high voltage standoff capability. By creating optimized crossed electrical and external magnetic fields around the vacuum envelope, we can generate a self-sustained Townsend discharge current which can be directly related to pressure. This technique is similar to the cold cathode gauge first developed by Penning, which was later modified by Redhead into the inverted magnetron gauge. In a typical cold cathode gauge the electrodes are a cylindrical design, with coaxial symmetry, which enables application of a uniform cross magnetic field. In many vacuum devices this coaxial electrode symmetry is not available. We overcome this obstacle by applying a ring-shaped magnetron magnet to the cathode electrode of a sealed vacuum device. In this arrangement the magnetic fields are not uniform, but the electron paths can still be significantly increased and even trapped resulting in enough ionization for a sustained discharge. The experimental technique to measure the time dependent pressure inside sealed vacuum

devices non-destructively will be discussed. The novel concept of this technique is that we use the existing vacuum device as its own measurement gauge, with only the application of external fields and instrumentation. We have experimentally demonstrated a measurement range of 10⁻⁶ to 10⁻² torr. A computer model of electron paths with some simple electrode geometries has been developed from which basic design guidelines can be derived. The model uses the Aleph finite-element particle-in-cell code developed at Sandia National Laboratories.

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000

11:40am **VT-MoM11 Vacuum Gauge Operation in Noisy Accelerator Environments**, *L. Smart*, Brookhaven National Laboratory

In the BNL Collider-Accelerator vacuum systems, many gauges are installed near subsystems that present challenges to obtaining noise-free gauge readings. Superconducting magnets, beam diagnostics & control elements, and even sunlight are some of the major contributors to nuisance variations in indicated pressure. Sometimes tried-and-true abatement techniques cannot be implemented, so compromises must be made. Observations and mitigation solutions for hot cathode, cold cathode, and convection-pirani gauges will be presented.

Monday Afternoon, October 29, 2012

Vacuum Technology

Room: 14 - Session VT-MoA

Gas Flow, Leaks, Permeation and Mass Analysis

Moderator: J.A. Fedchak, National Institute of Standards and Technology

2:00pm **VT-MoA1 First International Comparison of Standard Leak Calibrations of Metrological Institutes.** *K. Jousten*, PTB, Germany, *K. Arai*, NMIJ, Japan, *U. Becker*, *O. Bodnar*, PTB, Germany, *F. Boineau*, LNE, France, *J.A. Fedchak*, NIST, *V. Gorobey*, VNIIM, Russian Federation, *W. Jian*, SPRING, Singapore, *D. Mari*, INRIM, Italy, *P. Mohan*, NPL/I, India, *J. Setina*, IMT, Slovenia, *B. Toman*, NIST, *M. Vicar*, CMI, Czech Republic, *YH. Yan*, NIM, China **INVITED**

The measurement of leak rates has become an important test in industry for function tests, quality and safety management, and for environmental protection. Leak tests are performed not only for vacuum chambers, but also for various containers like electrical high power switches, pace makers, refrigerating systems, isolation vacuum, rims, and tanks. The leak tests are performed by leak detectors which mainly use helium as test gas. Traceability to the SI units is given by calibrated standard leaks that emit a well known flow rate of typically helium gas. Many National Metrological Institutes (NMIs) provide such traceability in their vacuum sections. The NMIs that signed the mutual recognition arrangement committed themselves to prove equivalence of their calibration measurement capabilities. The test of equivalence is a comparison where a transfer standard is calibrated at the participants' facilities and the results compared. The difference between the laboratory result and a reference value or a bias of a laboratory must not exceed the uncertainty of this difference or the bias. To test equivalence of standard leak calibrations NMIs from 11 countries performed a comparison with two helium permeation leaks as transfer standards. The leak rates were 4E-11 mol/s for standard leak L1 (1E-4 Pa L/s at 23°C) and 8E-14 mol/s (2E-7 Pa L/s at 23°C) for L2 respectively. For the latter, only 6 NMIs had measurement capabilities. Since leak rates from permeation leaks decrease with time, special evaluation procedures had to be applied to calculate a reference value and to compare the results of the NMIs. Also a statistical method was applied to evaluate a possible bias of a laboratory. Most of the 11 laboratories proved equivalent in the case of transfer standard L1 and all for L2. These results will be published and serve as basis for mutual recognition of calibration results.

2:40pm **VT-MoA3 Comparison of the Flow Ratio of the Permeation Type Helium Standard Leaks and the Gas Flow Generator Composed with a Small Conductance Element Made by Sintered Stainless Steel.** *N. Takahashi*, ULVAC Inc., Japan, *H. Yoshida*, AIST Japan

The possibility of the gas generator with a small conductance element made by sintered stainless steel as a small gas flow standard is examined.

Helium leak test is one of an important way for the non-destructive testing. Helium standard leaks are usually used for the calibration and for the adjustment of the sensitivity of helium leak detectors. Characteristic and calibration of the helium standard leaks were well studied from late 1980's to middle 1990's (1-6). However, the flow ratio of the one unit of standard leak is fixed. Helium flow ratio by the permeation through glass element usually shows large temperature dependence.

On the other hand, the gas flow generator composed with a small conductance element made by sintered stainless steel was reported as a variable, stable and wide range gas flow generator for the vacuum gauge calibration (7). We examined and made comparison of the helium gas flow ratio between the gas generator and the permeation type helium standard leaks. The flow ratios of the permeation type helium standard leaks are traceable to AIST/NMIJ. The relation of the helium gas flow generated by the generator and the permeation leaks was good agreement with less than 5% of difference. The generator generates wide range of helium gas flow less than 10⁻⁵ Pa m³/s. This experiment was performed on the ultra high vacuum equipment, which residual pressure was lower than 10⁻⁷ Pa, with a quadrupole mass spectrometer. The generator is also useful for the wide range, stable and easy way of calibration of standard leaks. Stability, conformity of the generator is also reported.

1) L. E. Bergquist, et al., *JVST A* **7**, 1989, 2414.

2) M. V. Iverson, et al., *JVST* **20**, 1982, 982.

3) J. M. Ball, *JVST A* **6**, 1988, 2860.

4) W. G. Bley, *Vacuum* **41**, 1990, 1863.

5) P. J. Abbott, et al., *JVST A* **14**, 1996, 1242.

6) C. D. Ehrlich, et al., *JVST A* **10**, 1992, 1.

7) H. Yoshida, et al., *Vacuum* **86**, 2012, 838.

3:00pm **VT-MoA4 Porous Plug Made of Sintered Stainless Steel used as Standard Conductance Element.** *H. Yoshida*, *K. Arai*, *T. Kobata*, National Institute of AIST, Japan

A porous plug made of sintered stainless steel, which is named as standard conductance element (SCE), has been developed as an open-type standard leak element for in-situ calibration of ionization gauges (IGs) and partial pressure analyzers [1]. Since the pore size of sintered filter is less than 1 micro meter, the molecular flow condition is realized up to 10 kPa of the upstream pressure of SCE. Therefore, four useful characteristics shown in below are available. (1) Flow rate is proportional to the upstream pressure of SCE. (2) Introducing various gas species with known flow rate is available by applying this single leak element. (3) Calibration using mixture gases is available. (4) Dependence of flow rate on the temperature is small and easy to compensate. In addition, the molecular conductance C_S of SCE has good long-term stability of less than 3 %/year typically. No significant influence is observed by introducing water vapor and bake-out. Since SCE with C_S from 1×10^{-10} m³/s to 2×10^{-9} m³/s is available, the flow rate of less than around 10^{-5} Pa m³/s is generated with various gas species.

Three typical applications of SCE are introduced. First is the quantization of the gas desorption rate for thermal desorption spectroscopy (TDS) [2]. The gas desorption rate was measured by a quadrupole mass spectrometer (QMS) which was calibrated by SCE with H₂, H₂O, N₂, CO, and CO₂ gases. Second is the measurement of the effective pumping speed S_{eff} of cryopump in the range from 10^9 Pa to 10^6 Pa [3]. H₂, CH₄, N₂, and Ar gases with known flow rate Q were introduced into the test chamber with the cryopump through SCE, and measured the pressure increment p by extreme high vacuum gauges. Pressure indications of the gauges were compensated by relative sensitivity factors for N₂. The last one is the in-situ calibration of IG and QMS. The standard pressure p was obtained from Q divided by S_{eff} , where Q and S_{eff} were determined by using SCE and conductance modulation method, respectively. The sensitivity of IG and QMS were measured for H₂, He, CH₄, H₂O, Ne, CO, N₂, C₂H₄, C₂H₆, O₂, Ar, C₃H₆, CO₂, N₂O, and C₃H₈.

These results show that SCE seems to satisfy almost all of requirements for the quantitative measurements in high and ultrahigh vacuum. SCE will be used as a new vacuum standard device.

[1] H. Yoshida, et al., *Vacuum* **86** (2012) 838-842.

[2] S. Inayoshi, et al., 52th Annual Symposium of the Vacuum Society of Japan (AVSSJ-52), Tokyo, Japan, 2011.

[3] M. Yamamoto, et al., Proceedings of international particle accelerator conference (IPAC2011), San Sebastian, Spain, 2011, 979-981.

3:40pm **VT-MoA6 Measurement of Gas Transport in Solids by a Saturation/Outgassing Method.** *L. Wang*, *J.A. Tanski*, *R.Y. Weinberg*, Los Alamos National Laboratory

Common flow techniques for measuring gas transport properties in solids requires the formation of a membrane which serves as a barrier to the movement of gas and the formation of leak-tight seal so the gas is diffusing through the membrane. However, for many materials, especially brittle non-metallic ionic solids, meeting these two requirements are difficult. In this study, an alternative method based on first saturating the material with the gas at a known pressure and subsequently allowing the absorbed gas to diffuse out to a static vacuum environment was developed for measuring gas transport properties of this type of materials. The method uses a sample of a well-defined geometry, in this case a right cylinder, and a computer program (DiSol) was developed to model the desorption process and extract diffusivity and solubility from the measured outgassing curve. To demonstrate the viability of the method, helium transport in a high-density polyethylene (HDPE) cylinder was measured, and the results obtained compared well with the published diffusivity and solubility values. The details of the method and the HDPE data obtained in this study will be presented.

4:00pm **VT-MoA7 Report on Workshop on Measurement Characteristics and Use of Quadrupole Mass Spectrometers for Vacuum Applications**, *K. Jousten*, Physikalisch Technische Bundesanstalt, Germany, *J. Šetina*, Institute of Metals and Technology, Slovenia, *R. Ellefson*, REVac Consulting

The ISO Technical Committee (TC) 112 is responsible for international standards in the field of vacuum technology. Supporting TC112 are three working groups (WG): WG1 is responsible for vacuum pumps, WG 2 for vacuum instrumentation and WG 3 for vacuum hardware. Hence, the WG 2 is responsible for total and partial pressure measurement in vacuum. In 2006, WG 2 began a project towards standardization for the specification and calibration of quadrupole mass spectrometers (QMS). A new ISO (Draft) standard 14291 "Definitions and specifications for quadrupole mass spectrometers" will be published in 2012. Additionally WG 2 is working on documenting the proper use of a QMS and establish a standard for meaningful calibration procedures. In support of this ISO goal, the European Metrology Research Programme has established a project IND12 "Vacuum metrology for production environments" to open new measurement capabilities for vacuum and to help industry to characterize vacuum in industrial environments.

One of the focuses of this IND12 effort is to address traceability to national measurement standards for partial pressure measurements and outgassing rate measurements for materials characterization in industry. To gather current status on such methods, a workshop on calibration of the QMS for industrial use was held in Bled, Slovenia. Quadrupole mass spectrometers are widely used to measure partial pressures in vacuum although the performance and accuracy of these measurements is the subject of ongoing discussion among users. The wide range of partial pressure measurement needed from UHV/XHV (10^{-10} Pa) to 1 Pa emphasizes that one type of QMS does not address all applications. From presentations at this workshop, application-specific data on performance of QMS types gives guidance for selection of a QMS type for an application. Talks were given on setup and ion source operation recommendations; initial calibration methods for an application and *in situ* calibration methods for verification or recalibration of a QMS during use. The fact that QMS electron energies differ by application and manufacturer leads to the requirement that gas species calibration is needed for each QMS for accurate partial pressure or compositional analysis. Papers on the electron-ion space charge at pressures $>10^{-4}$ Pa in ion sources were given together with gas interference where addition of a partial pressure of a new chemical species B gives a different ion current output for species A (when A is known to be at its original partial pressure). Results of these talks at the workshop will be summarized in this presentation.

4:20pm **VT-MoA8 Mass Spectrometry a Mile Deep: Issues and Solutions for Underwater Vacuum Systems**, *R.T. Short*, SRI International **INVITED**

There are numerous advantages to performing *in situ* chemical analyses in the field rather than collecting samples for laboratory analysis. These include reducing the possibility for sample contamination or degradation, significantly improving the spatial and temporal resolution of analyses, and providing the ability to implement adaptive sampling strategies by receiving the analytical information in real-time or near real-time. Many analytical instruments, such as mass spectrometers (MSs), require high vacuum for operation. Vacuum systems for these portable instruments must be reasonably small, low-power, and often more rugged than needed for laboratory operation. Underwater instruments of this type have the additional requirements that the vacuum systems must be self-contained (or exhaust to a high-pressure environment) and the sampling interfaces must withstand pressure differentials greater than 1 atmosphere – hydrostatic pressure increases by approximately 1 bar for every 10 m depth in the ocean. Consequently, all of these constraints must be taken into account when selecting or building vacuum systems for these applications.

Several groups around the world have been working on developing and using underwater MSs for *in situ* chemical measurements in oceans, lakes, and rivers – at times over a mile deep. To date, all of these operational underwater MSs have employed membrane-inlet interfaces to introduce analytes into the vacuum of the MS. Analytes that are soluble in the membrane material (typically polydimethylsiloxane or Teflon) permeate through the membrane and evaporate into the ion source of the MS, where they are ionized by an electron impact source. For operation at depth, the membrane must be supported by a porous frit to withstand the increased hydrostatic pressure. Membrane inlet MSs are very effective at detecting and quantifying dissolved gases, light hydrocarbons, and volatile organic compounds, often at trace levels.

Design considerations for underwater MSs will be discussed in this presentation, along with examples of specific components chosen for the vacuum systems. In particular, the focus will be on problems with operating vacuum systems under these often extreme and harsh conditions, along with solutions to mitigate these problems. In addition, we will present examples

of deployments of underwater MSs for a variety of applications on a number of unmanned deployment platforms.

5:00pm **VT-MoA10 Performance Optimization for Autoresonant Ion Trap Mass Spectrometers**, *G.A. Brucker*, *J. Rathbone*, *B. Horvath*, Brooks Automation, Inc., Granville-Phillips Products

A new generation of residual gas analyzers based on autoresonant ion trap mass spectrometry (ARTMS) technology has recently become commercially available and is rapidly gaining market acceptance for vacuum processing applications. Recent investigations into this novel ion trap technology have focused on a detailed understanding of the role of ion generation and storage on the mass spectrometer's performance. The rate of ion formation, the initial energetics of the ions stored inside the trap, the shape of the electrostatic trapping potential and the characteristics of the radiofrequency signal sweep used to eject the ions all have a significant impact on the performance of an ART MS instrument. Unit-to-unit variations in ion formation rates and energetics due to mechanical assembly tolerances, can lead to slight variations in ion trap performance which can be easily corrected through adjustment of the electron beam focusing optics and the shape of the electrostatic trapping potential. This presentation describes a straight forward methodology that has been developed in our research laboratory to manually optimize ion trap performance during routine measurements and also an automated tuning procedure (Auto-Tune) that is built into the control software and can be used to restore trap performance each time an ART MS sensor is fitted with a new filament assembly.

5:20pm **VT-MoA11 Differential Pumping Method for a Fast Partial Pressure Analyzer of Recent Design to Extend its Upper Pressure Limit Up to the Torr Range**, *P.C. Arnold*, *T.C. Swinney*, Brooks Automation, Inc., Granville-Phillips Products

The upper pressure limit of a partial pressure analyzer (PPA) was extended by differential pumping so that gas composition of process pressures are evaluated. The selection of orifice size for differential pumping, first by calculation, then by empirical data, is presented. The range of a linear relation for reduced total pressure vs. process pressures in the millitorr range for several gases is presented. The validity for scans of multiple gas species for 0 – 130 amu was also evaluated by testing, comparing scans in the high pressure region to those in the reduced pressure region. The application of this method for an autoresonant ion trap will be presented for the first time in this pressure reduction protocol as it provides measurement of fast transients at these higher pressures. Trade-offs for orifice sizes and pressure reduction ratios will be discussed. The test set-up as it relates to the AVS Recommended Practices and the conclusions for pressure ranges that it provides will be given.

Vacuum Technology Room: 14 - Session VT-TuM

Pumping, Gas Dynamics and Modeling Moderator: L. Wang, Los Alamos National Laboratory

8:00am **VT-TuM1 Gas Dynamics Modelling for Particle Accelerators, O.B. Malyshev**, STFC Daresbury Laboratory, UK **INVITED**

Design of accelerator vacuum chamber requires an input from different scientific disciplines such as surface science, material science, gas dynamics, particle beam dynamics, and many others. Although vacuum scientists work on the boundary field between these disciplines the gas dynamics is one that allows jointing all these to the vacuum science for particle accelerators.

The particle accelerator requirement to vacuum defined by beam gas interactions that should be negligible comparing to the other phenomena and effects limiting the quality of the beam, so these requirements are in free molecular regimes: HV, UHV or even XHV. At such low pressures the main source of gas in the vacuum chamber is molecular desorption from materials used for vacuum chamber and in-vacuum components.

The outgassing rates depends on material, its cleaning procedure, treatments (polishing, etching, coatings, bakeout, etc.), time in vacuum, irradiation or bombardment by particles (photons, electrons, ions, etc.) and accumulated irradiation dose. Therefore, the outgassing rates vary in very wide range.

The gas dynamic is used to design the research facilities to accurately measure and to study outgassing rates at different conditions, then it used for data analysis. By applying these data to the accelerator vacuum design one have to consider that outgassing is often non-uniform and changes with time with different functions. Full 3D modelling is possible with TPMC codes, however, it is time consuming work and not ideal for pumping and design optimization, so it is used for components or for finalized design. Meanwhile, during the optimization study the most time-efficient way is using 1D diffusion model where all parameters are defined as a function of longitudinal coordinate (along the beam path).

The examples accelerator vacuum chamber designer should also consider such effects as thermal outgassing, photon, electron and ion stimulated desorption, beam induced electron multipacting and ion induced pressure instability.

8:40am **VT-TuM3 Transient Flow of Rarefied Gas through a Short Tube, F. Sharipov**, Federal University of Parana, Brazil

Steady flows of rarefied gases through orifices, slits, short tubes, and channels are well studied. In spite of the high practical interests to the transient flows of rarefied gases, the problem of short tube flow has not been studied from this viewpoint. The aim of the present work is to study transient rarefied gas flow through a short tube on the basis of the direct simulation Monte Carlo method. The mass flow rate and flow field are calculated as a function of the time in the transitional and hydrodynamic regimes with respect to the gas rarefaction. Two values of the pressure ratio, i.e., 0.1 and 0.5, and two values of the aspect ratio, i.e. 1 and 5, are considered. A characteristic time equal to that needed to cross the tube radius with the most probable molecular speed is introduced. The typical time to establish the stationary flow is calculated. The flow field past the tube reaches the steady state during the same time in the transitional regime and it takes a longer time in the hydrodynamic regime.

9:00am **VT-TuM4 Experimental Results and Direct Simulation Monte Carlo Modelling of a High-Performance Large-Scale Cryopump, S. Varoutis, Chr. Day, X. Luo, H. Haas**, Karlsruhe Institute of Technology, Germany, **F. Sharipov**, Federal University of Parana, Brazil

The main duty of the vacuum pumping system of fusion devices is to pump out the fusion exhaust gas. Due to the fact that very high throughputs have to be coped with, large pumping speeds are required. This is typically provided by cryogenic pumping, supplied with cryogen medium at 4 K and 80 K. The concept for the cryosorption vacuum system of ITER, the next generation fusion experiment currently being built in Europe, has been developed at the Karlsruhe Institute of Technology (KIT). As a result to the large gas flows, which are unusually high for a cryopump, the pumps are operated in the transitional regime. A further development and improvement of the system requires a corresponding numerical modelling of the gas flow inside the pump housing and near the cryopanel section.

The aim of the present work consists of the computational investigation of a 2D axisymmetric complex geometry of a model cryopump by the Direct Simulation Monte Carlo (DSMC) method. Since the flow close to the

cryopanel can be assumed free molecular due to low pressure levels, the capture coefficient of the cryopanel can be estimated by applying the Test Particle Monte Carlo method. Then, this information can be used as input data to the corresponding DSMC simulations. The macroscopic parameters of practical interest as the bulk velocity, the pressure and the temperature in the whole flow field, have been calculated as a function of the incoming gas throughput and of the pump inlet valve position. Furthermore, the present numerical results have been thoroughly compared with corresponding experimental results obtained at KIT for the case of an ITER model cryopump.

The importance of these calculations is based on the fact that they can provide information for quantities which are not accessible for measurement during pump operation and that they can be used for prediction of the pump behavior. This paper describes a post-operational investigation of a built pump and is thought as a proof-of-principle test to include this approach in the design process of a future pump development.

9:20am **VT-TuM5 Development of a PhD-level course in Vacuum Science and Technology, P. Eklund**, Linköping University, Sweden

There are many available courses in Vacuum Technology, including those offered by the AVS. They typically have an engineering-oriented approach aimed at the practical user of vacuum system. When I started teaching a PhD-level course in Vacuum Science and Technology, I was faced with a different – pedagogical and scientific – challenge. The attendees are PhD students who work, or will work, with vacuum in their PhD research and future careers, but most are not vacuum practitioners per se. Neither is the teacher. In such a course, the students need to achieve an in-depth understanding of the science of vacuum and how it can ultimately affect their research. Here, available textbooks on the topic of “Vacuum Technology” are not at PhD level – they tend to be “engineering user’s guides” or similar.

In achieving this, a clear definition of the objectives is essential. In a PhD course, the aim is to give students a thorough understanding of how vacuum components and vacuum systems work, and the fundamental physics and chemistry behind them – emphasizing the latter part, in contrast to a more engineering-oriented course. Among others, this means that students should understand and be able to define the vacuum concepts (ideal, rough, low, high, ultrahigh, etc...), understand and be able to explain in own words the kinetic theory of gases, the principles for gas flow at low pressures, and physico-chemical phenomena in vacuum (evaporation, condensation, solubility, permeation, adsorption, absorption, desorption). They should also be able to apply the knowledge and understanding listed above to practically and theoretically relevant situations in vacuum science and technology, communicate this understanding orally and in writing, and be able to critically reflect on scientific articles relevant to vacuum science and technology.

Here, I will discuss my pedagogical and scientific approach to such a course and how to align the course activities for the PhD students to reach the above goals. I will also discuss the examination format, and why I have found it to be particularly suitable for this type of course. It is a combination of continuous examination in connection with the lectures and home examination, which also contains an assignment connecting the course content to the laboratory work of the students. The continuous examination is not mandatory, but gives credits for the home examination. The mandatory parts of the course are examined through peer review and an ending seminar.

10:40am **VT-TuM9 A Comparison between Numerical and Analytical Models of Turbomolecular Drag Pump’s Stages, I.F. Cozza**, Agilent Technologies, Italy, **M. Rose**, PI-DSMC, Germany, **R. Arpa, H. Telib**, Optimad Engineering S.R.L., Italy

The design and optimization of vacuum pumps requires a deep knowledge of the internal gas-dynamics, and a large trial-and-error process to fix the design parameters. A common practice in the TMDP’s industry is to perform an experimental design of new pumps, by testing the global performances of prototypes. This approach has two drawbacks: it doesn’t give indications on the details of the internal fluid dynamics of the problem, and the design and realization of prototypes has significant costs and requires long times, that could affect the time-to-market of new products.

In this framework, an accurate and efficient numerical analysis tool could meet these needs. This tool should model the tridimensional, local flow features, such as pump leakage and development of the rarefied gas flow along the curved channels, and take into account the inertial forces.

In this work, two approaches will be presented: a full 3D Direct Simulation Monte Carlo numerical analysis of the Siegbahn drag stages, and a semi-

analytical approach based on the numerical solution of the Linearized Boltzmann Equations.

The DSMC simulations have been performed using a DSMC software package called PI-DSMC. The sampling and collision cells were generated automatically from a triangular mesh describing the shape of the solid body. The reflection of molecules by the walls was investigated to choose the proper particle/surface interaction model. The temperature of the rotor, the stator and the gas at the inlet have a fixed value. The effect of rotor temperature on the performances of the stage has been investigated. The collisions between nitrogen and hydrogen molecules were modeled using the variable hard sphere model with the common parameters.

The semi-analytic model is developed for steady flows in spiral molecular drag stages, and it is based on the solution of the Boltzmann Equation (BE) with a BGK closure. The order of the original problem is reduced in the physical space to 2D, by introducing assumption of locally known flow development of the distribution function along the spiral channel. Thus, 2D-BE calculations of the flow rates and stresses will be performed in a finite number of sections, suitably positioned along the spiral channel, from the outlet to the inlet, in order to recover the integral performances of the pump. The original BGK equation is linearized in the most significant parameters (rotational speed and pressure gradients), and solved in the reference cross-section, by means of a DVM scheme. Maxwell diffuse boundary conditions and impermeability are provided at walls. The local values of pressure and torque are obtained consistently by enforcing the mass flow.

11:00am VT-TuM10 Improved Modelling and Measurement of the Rotor Temperature of Turbo-Molecular Pumps in Magnetic Fields. A. Jansen, KIT, IEKP, Germany, N. Kernert, KIT, IKP, Germany, J. Wolf, KIT, IEKP, Germany

When designing a vacuum system with turbo-molecular pumps (TMP) in an external magnetic field, one needs to know the influence of eddy currents on the rotor temperature to ensure safe operating conditions. The KATRIN neutrino experiment will operate about 20 TMPs in the vicinity of superconducting magnets, pumping out tritium gas from the electron beam-line of the experiment. In a dedicated test setup with Helmholtz coils systematic studies have been conducted, investigating the rotor temperature and stability for TMPs with magnetic and ceramic bearings at full speed. The rotor temperature was monitored with an infra-red pyrometer as a function of gas load, magnetic field strength and direction of the field. For interpretation of the data and predictions for different operating conditions an empirical model has been developed, describing the rotor temperature versus time as a function of gas flow and magnetic field, using 5 pump-specific parameters, which characterize heating effects of eddy currents and gas friction as well as cooling by radiation loss and convection. Since this approach assumes a homogeneous field, we extended the model, replacing the field and friction terms by the motor current of the pump, which compensates the retarding effects of eddy currents and gas flow. This new model can now be used for inhomogeneous magnetic fields.

KATRIN is supported by the German BMBF project 05A11VK3, the Helmholtz Alliance Astroparticle Physics and HGF.

11:20am VT-TuM11 Test of Temperature-Dependent NEG Activation and Stability of Gold-Plating in the KATRIN Experiment. W. Gil, L. Bornschein, J. Wolf, Karlsruhe Institute of Technology, Germany

The Karlsruhe Tritium Neutrino (KATRIN) experiment will measure the neutrino mass with an unprecedented sensitivity of $0.2 \text{ eV}/c^2$ by investigating β -electrons from tritium decay. While the electrons are magnetically guided through a beam tube to the spectrometer by superconducting solenoids, the tritium flow rate from the source has to be reduced by at least 14 orders of magnitude by differential pumping and cryo-sorption. The last stage of the pumping section is the 7 m long cryogenic pumping section (CPS), using a pre-condensed argon-frost layer to capture tritium molecules. For reducing the adsorption of tritium on the wall of the beam line and for more efficient regeneration of the cryo-pump its inner surface has been gold-plated, using a standard industrial galvanic process. Non-evaporable getter (NEG) strips (SAES St707) will be installed inside the last meter of the beam tube of the CPS as a fallback system, protecting the spectrometer in case of a failure of the cryogenic system. The standard NEG activation temperature is 450°C for about 60 min, but can be reduced with prolonged activation time. However, the activation temperature in the CPS should be as low as possible with respect to the superconducting solenoids around the beam tube and the stability of the gold-plating. Therefore, a test has been set up, optimizing the activation temperature and time with regard to the NEG's pumping efficiency, the stability of the gold-plating, and the safety of the magnets. This paper presents results of the temperature-dependent NEG activation efficiency and the influence on the gold-plating.

11:40am VT-TuM12 Improving the Pump Down of UHV Systems by the Additional Pumping Speed Provided by NEG Pumps. F. Siviero, A. Bonucci, A. Conte, L. Caruso, L. Viale, P. Manini, SAES Getters, Italy

The study of pump down processes is one of the basic topics of vacuum technology since its early days. Its relevance from the practical point of view is very high in a variety of systems including large machines like accelerators, surface science equipment, scanning/transmission electron microscopes and many other analytical systems and sealed off devices. At present the bake-out of these systems may require days to weeks, resulting in a considerable use of time and energy. Here we report on a series of experiments aimed at investigating how an increase of the total pumping speed during the pump down influences the behaviour of the main gases of interest, i.e. water and hydrogen. Several pumping configurations are compared, including turbo molecular pumps, large sputter ion pumps (SIP), Non Evaporable Getter (NEG) and a new SIP/NEG combination pump called NEX Torr[®] [1]. The most relevant desorption models and their predictions for the pump down processes are expressed in an explicit form and compared with the experiments. The results of the study confirm that water desorption can largely benefit from an increase of the available pumping speed, due to the reversible nature of its adsorption kinetics. As far as hydrogen is concerned, a higher pumping speed at the end of the bake provides a lower partial pressure. This translates into the possibility of either reducing the duration of the bake-out process or improving the ultimate achievable vacuum, both issues having practical interest in vacuum systems for research and industrial applications.

[1] NEX Torr is an International Trademark registered by the "Madrid System" property of SAES Getters S.p.A.

Tuesday Afternoon, October 30, 2012

Vacuum Technology

Room: 14 - Session VT-TuA

Accelerator and Ultra-Clean Vacuum Systems

Moderator: L. Smart, Brookhaven National Laboratory

2:00pm **VT-TuA1 Design of the Vacuum System for the SuperKEKB Positron Ring**, *Y. Suetsugu, K. Kanazawa, K. Shibata, T. Ishibashi, H. Hisamatsu, M. Shirai, S. Terui*, High Energy Accelerator Research Organization, Japan **INVITED**

A two-ring electron-positron collider with asymmetric energies, the SuperKEKB, has been designed as an upgrade of the KEKB B-factory (KEKB). The SuperKEKB aims for a maximum luminosity of $8 \times 10^{35} \text{ cm}^{-2} \text{ s}^{-1}$, which is approximately 40 times larger than that of the KEKB. The upgrade of the vacuum system is a key factor that will allow the SuperKEKB to achieve unprecedented high performance. As for the positron ring, most of the beam pipes are newly designed to reduce beam impedance and, especially, to manage the electron cloud effect (ECE), which is essential to keep the low-emittance beam stable. The beam pipes basically have antechambers at the both sides of a beam channel. Various vacuum components adaptable to the antechamber scheme with low beam impedance and high thermal strength had been developed. The bellows chambers, for example, have a comb-type RF-shield with the same cross section to the beam pipe, and the main vacuum pump consisting of NEG strips is inserted into one of the antechambers. The antechamber scheme is also effective to mitigate the ECE, that is, it structurally suppresses the photoelectron effect. A side wall of the antechamber, where the synchrotron radiation hit directly, is roughened so as to reduce photon reflections. In order to reduce secondary electron effect, on the other hand, the inner surface of beam channel is coated with titanium nitride (TiN). Furthermore, the longitudinal grooved surface and the clearing electrode are prepared for the beam pipes in dipole magnets and the wiggler magnets in the ring, respectively. In addition, the beam pipes in drift spaces are wound by solenoid coils. These mitigation techniques are the fruits of various theoretical and experimental studies so far. The SuperKEKB positron ring is the first one that will adopt these techniques in a large scale. The design of vacuum system has been mostly completed, and the mass production of beam pipes has started. The vacuum system design and some key issues for SuperKEKB positron ring will be reported here together with the present status.

2:40pm **VT-TuA3 Status of the FRIB Driver Linac Vacuum Calculations**, *B. Durickovic, P. Gibson, P. Guetschow*, Michigan State University, *R. Kersevan*, CERN, Switzerland, *D. Leitner, M. Leitner, L. Lingy, F. Marti, G. Morgan, M. Schein, M. Shupiar*, Michigan State University

The Facility for Rare Isotope Beams (FRIB) is a heavy ion fragmentation facility to produce rare isotopes far from stability for low energy nuclear science. The facility will utilize a high-intensity, superconducting heavy-ion driver linac to provide stable ion beams from protons to uranium at energies greater than 200 MeV/u and at a beam power of up to 400 kW. The beam will be fragmented on a multilayer high power fragmentation target and separated in a high resolution fragment separator.

Two ECR ion source injectors will provide highly charged ions for the superconducting linac for efficient acceleration. In order to transport the heavy ions at the low velocities of the injection beam the vacuum systems need to be carefully designed to avoid beam losses due to charge exchange. For uranium $33+$, for example (one of the commissioning beams), the cross-section for electron capture from the residual gas is so large at low energies ($\sim 12 \text{ keV/u}$ in LEBT) that a residual gas pressure of 10^{-6} Torr would lead to unacceptable beam losses in the analyzing magnet.

Similarly, in the warm section of the superconducting linac, beam losses due to interaction of the beam with residual gas need to be minimized in order to keep the average uncontrolled beam loss well below 1 W/m as required for maintainability of the accelerator and safety considerations.

These beam loss requirements, as well as the need for managing vacuum levels in high loss regions such as beam stripping and collimation areas, led to the establishment of minimum baseline vacuum requirements for all areas of the accelerator system. In addition, the SRF cavities must be protected from contamination that could possibly migrate from the stripper region, collimator systems, or target systems.

CAD vacuum models of each area are made based upon the accelerator lattice file, and Monte Carlo simulations of vacuum levels are performed using MolFlow+ to help determine or validate the vacuum hardware

configuration needed to meet the baseline requirements. This talk will describe the FRIB facility vacuum requirements, and report on the methods and status of the FRIB vacuum calculations.

This material is based upon work supported by the U.S. Department of Energy Office of Science under Cooperative Agreement DE-SC0000661

4:00pm **VT-TuA7 Contamination Control and Cleaning Techniques for Ultra Clean Vacuum Systems**, *H.G.C. Werij, N.B. Koster, J.C.J. van der Donck, A.J. Storm, R. Verberk, R. Versluis*, TNO Technical Sciences, The Netherlands **INVITED**

Cleanliness requirements in high-tech science and industry are getting more and more challenging. At the same time, the consequence of not properly adhering to these requirements could be devastating. Whereas the general way of working involves prevention, inspection/monitoring and cleaning, each particular application requires its own dedicated systems approach. For instance, is it particulate or molecular contamination we have to deal with? Do we really need an ultrahigh vacuum or should it be ultraclean with respect to certain contaminants? And what is the trade-off between ultraclean production and in-situ cleaning? Such questions have to be answered in order to find the solutions that are both adequate and cost-effective at the same time.

In the presentation several examples will be addressed, including delicate space instrumentation, (extreme ultraviolet) lithography tools and equipment for fusion energy (ITER). We will present how contamination should be dealt with in the design phase, which not only involves choice of materials, but also geometrical layout and handling. We will show that for certain applications the traditional approach of using ultra-high vacuum might not yield the optimum result. By introducing low-pressure ultraclean gases combined with differential pumping both molecular and particulate contaminants can be mitigated very efficiently, as is demonstrated in numerous experiments.

As far as inspection and monitoring is concerned, we will give a brief overview of several sensing technologies, currently being developed at TNO. These consist of ionization sensors and optical sensors, the latter being integrated nanophotonics sensors, which may be used to detect molecular contaminants. For particle detection down to 50 nm diameter an automated particle scanner has been developed, which will be touched upon as well.

In the field of cleaning, apart from more traditional methods like wet, CO_2 , and ultrasonic cleaning, we will address plasma cleaning technology developed in-house. We will show experimental results obtained using our shielded microwave induced remote plasma setup and hydrogen radicals.

4:40pm **VT-TuA9 Large Thermal Vacuum Chamber for TB/TV Tests and Optical Calibration of Space Instrumentation**, *R. Versluis, R. Verberk, E.C. Fritz, W.L.M. Gielesen*, TNO Science and Industry, The Netherlands

Part of the TNO Space activities is the development and qualification of Optical Instruments for Space Applications. Especially the Earth Viewing Spectrographs constitute a significant part of the calibrations. Before these instruments will be launched into orbit they undergo a series of qualification tests, which typically consist of:

Performance test (PF)

Vibration test (VB)

Thermal balance/thermal vacuum test (TB/TV)

Performance test (PF)

Optical Calibration (OC)

TNO is building a Thermal Vacuum Chamber (TVC) in one of the TNO cleanrooms that can perform all of these tests (except the vibration tests) on these instruments. In this talk we will present an overview of the design, engineering, manufacturing and qualification activities that are related to this thermal vacuum chamber. The TVC will have an internal free volume of about 72 m^3 . The complete internal surface area of 100 m^2 will be entirely covered with a cooling shroud with temperature control between 95 K and 373 K. The required shroud temperature stability is less than 1 K_{pp} , the required steady state spatial homogeneity better than 5 K_{pp} . In order to reach low pump down times and allow testing of space instruments with relatively high outgassing rates, the required pumping capacity is between 5000 and 10000 L/s. Furthermore, a very high level of cleanliness is required to protect the space instrument optical system and TVC viewports from contamination, which could decrease the optical transmission, especially in the UV wavelength range. Contamination prevention is especially important because the complete calibration test can last as long as a few months, during which the system will be at vacuum and deposition of

contamination can affect the optical throughput. The TVC will be equipped with a residual gas analyzer, cold finger and thermal quartz crystal microbalance to perform online and offline contamination monitoring and analysis.

For the Optical Calibration the Space instrument will be integrated in a cradle with two rotational degrees of freedom. With this the complete Field Of View of all optical ports of the instrument can be illuminated by optical stimuli placed outside the vacuum vessel.

The presentation will highlight specific issues related with this type of test facility, such as standard and emergency procedures for evacuating and venting the chamber during cryogenic operation. Product assurance issues such as contamination prevention of the test object and test object integrity. Minimising leak rates and outgassing of feedthroughs and stages, particle contamination prevention and other issues of the tests performed at high vacuum and low or high temperatures.

5:00pm **VT-TuA10 A Large Seismic Attenuation System in UHV, R. Takahashi**, National Astronomical Observatory of Japan, *Y. Saito*, High Energy Accelerator Research Organization, Japan

Interferometer gravitational wave detectors require an ultra-high vacuum chambers which the laser beams pass through. KAGRA, the large-scale cryogenic gravitational wave telescope in Kamioka, has two 3-km vacuum tubes kept in $\sim 10^{-7}$ Pa of vacuum pressure so as to reduce scattering-effects due to residual gas molecules.

The interferometers consist of high quality mirrors, which should be isolated from ground vibration strongly. The vibration isolation system needs not only attenuation more than 109 at 100Hz but also reduction of root mean square motion of the mirrors. Many kinds of mechanisms for isolation at low frequencies have been suggested for gravitational wave detectors. We employed an inverted pendulum and geometric anti-spring filters as the isolator in KAGRA. We found diamond-like carbon (DLC) coatings are suitable for reduction of scattered light around the mirrors. The coatings have low outgassing, low reflectivity, and low scattering loss.

5:20pm **VT-TuA11 Vacuum System of Cornell Energy-Recovery LINAC Prototype Injector, Y. Li, X. Liu, K.W. Smolenski, I. Bazarov, B.M. Dunham**, Cornell University

A prototype electron injector was designed, constructed and operated at CLASSE, as an important first step toward the Cornell ERL (Energy-Recovery LINAC) based synchrotron radiation facility. The injector is designed to generate average beam current up to 100-mA, and electron beam energy ranging 5-MeV to 15 MeV. Main features of the injector include a laser-driven photo-emission electron source, a cryo-module containing superconducting RF cavities, electron beam transport beamlines equipped with a suite of beam diagnostic instrument, and a 600-kW electron beam stop. Recently, significant milestones were reached for the prototype injector. Most noticeably, we have achieved an average beam current of 52-mA at a beam energy of 5-MeV from activated GaAs photo-cathodes, breaking a long-standing world record of 32-mA from a laser-driven photo-emission electron source. There are many challenges in vacuum system design for the prototype injector. It needs to provide an extremely-high vacuum (XHV) environment for the photo-cathodes, flexibilities in beam transport beamlines for development of beam instrumentation, as well as sufficient vacuum pumping capacity to handle very large dynamic gas-load at the beam stop. In the past 3+ years of operations, the injector vacuum system has performed satisfactorily. To confirm the pumping performance, we calculated pressure profile along the main transport beamlines during the high beam current runs, and calculated pressure profile agreed well with the measured pressure profile. In this paper, we describe the design and the operational experiences of the prototype injector vacuum system, and address remaining operational issues arising from high beam current operations.

5:40pm **VT-TuA12 Injection Vacuum System at the TPS, C.K. Chan, C.C. Chang, C.L. Chen, C.S. Yang, C. Chen, Y.H. Liu, K.H. Hsu, Y.T. Huang, H.P. Hsueh, S.N. Hsu, G.Y. Hsiung, J.R. Chen**, NSRRC, Taiwan, Republic of China

The Taiwan Photon Source (TPS) is a 3 GeV synchrotron facility and aimed to have a low emittance electron beam maintaining the top-up operation. A 12-m long TPS injection section contains four kicker ceramic chambers (K1~K4) and one out-of-vacuum injection septum to provide the stored beam a horizontal bump for beam injection off axis. The kickers (K2, K3) and injection septum are placed in an adjustable plate, which can provide a 5 mm displacement for the injected beam close to the stored beam so as to decrease the kicker strength. The construction of the injection section is

completed and we will describe the design, manufacturing process and some test results for the injection section.

Tuesday Afternoon Poster Sessions

Vacuum Technology

Room: Central Hall - Session VT-TuP

Vacuum Technology Poster Session and Student-built Vacuum System Poster Competition

VT-TuP2 Pressure Effects in Autoresonant Ion Trap Mass Spectrometers (ARTMS), P. Acomb, G.A. Brucker, J. Rathbone, Brooks Automation, Inc., Granville-Phillips Products

Autoresonant ion trap mass spectrometers (ARTMS) are gaining rapid acceptance in the vacuum market both as residual gas analyzers as well as process gas monitors. During the course of routine analysis, most ARTMS instruments are required to provide accurate analytical results over a wide range of total pressures and gas compositions. Since the performance of ion traps is pressure dependent, it is important to understand how total pressure impacts gas analysis results and to understand the methodologies available to adjust operational parameters based on present pressure conditions. Important performance specifications that are affected by total pressure conditions include baseline offset, resolving power, sensitivity, dynamic range and fragmentation patterns. The pressure-related physical phenomena presently known to impact ion trap performance are identified and associated to the specific pressure ranges at which they are relevant. The ion trap operational parameters available to the user to optimize trap performance as a function of total pressure are explained. Vacuum practitioners presently using ARTMS instruments for gas analysis can quickly, easily and dynamically apply the concepts introduced in this poster to optimize the performance of their gas monitors as a function of total pressure conditions.

VT-TuP3 Investigation of a Method for Measurement of Water Vapor Coverage on Technical Surfaces, M. Sefa*, Laboratory Lotric d.o.o., Slovenia, J. Šetina, Institute of Metals and Technology, Slovenia

Coverage of a surface by a gas can be determined by desorption method where amount of gas is calculated by integrating the released flux. This can be measured fairly straight forward in a case when desorbed gas don't re-adsorb on surfaces of measurement chamber. Then mass conservation laws are applicable and flux can be determined by measurement of a pressure drop across the orifice of known conductance. In the case of adsorbing gas, like water vapor, the flux through the orifice is not equal to the flux coming from the sample, because significant amount can be re-adsorbed by the surfaces of measurement chamber. To determine surface coverage of water vapor on technical surfaces we have developed a special procedure where adsorption and desorption on the surfaces of measurement chamber can be taken into account. The sample is exposed to water vapor at known pressure (in the range from 0.1 kPa to 2.5 kPa) in a preparation chamber. After equilibration of surface coverage with the gas phase, the preparation chamber is opened to a measurement chamber which is continuously pumped through a known conductance. In the case of water vapor the shape of pressure burst is significantly influenced in the beginning by adsorption and later by desorption in the measurement chamber. However, the time integral of pressure burst over sufficiently long period represents the amount of water vapor coming from preparation chamber. This amount is sum of gas phase, desorbed amount from surfaces of preparation chamber and desorbed amount from the sample surface. In the presentation the measurement system will be described and examples of water coverage measurements on surfaces of Cu, Ni, Al and stainless steel will be given.

VT-TuP4 Flow of a Binary Gas Mixture Into Vacuum: Experiment, Models, Simulation, M. Vukovic, Tokyo Electron, US Holdings, R. Johnsen, University of Pittsburgh

The flow of gas through a small orifice into vacuum was experimentally analyzed by Fujimoto & Usami (1984). They developed a formula to describe the flow conductance as function of the orifice rarefaction parameter. R. Johnsen and B. K. Chatterjee (2011) measured the flow rate of a minority gas (H_2 , O_2 , CO_2) in a carrier gas (He, Ar) using the University of Pittsburgh ion drift tube experiment. Using the hard-sphere collision potential they also developed a heuristic formula to model the minority gas conductance rate as function of the carrier gas rarefaction parameter. In this work we generalize their formula to arbitrary intermolecular potentials. We also develop an alternative model and formula that successfully describes the data. We will describe the ion-drift tube experiment, discuss the two models and formulas. We will also present

results of DSMC simulations using the Graem Bird's DS2V code (<http://www.gab.com.au>).

T. Fujimoto and M. Usami, ASME Trans. J. Fluids Eng. 106, 367 (1984).

R. Johnsen and B. K. Chatterjee J. Vac. Sci. Technol. A 29, 011002-1 (2011)

VT-TuP5 A Calibration System for Helium Leak Calibrator, Y.-W. Lin, C.-C. Hung, C.-P. Lin, C.-N. Hsiao, F.-Z. Chen, Instrument Technology Research Center, Taiwan

A calibration system for helium leak calibrator was developed, and the measurement uncertainty associated with the system. The design of the system took into consideration of influencing factors that include reference standard leak calibrator (ref.), leak calibrator under testing (dut.), vacuum chamber, He leak detector, I/O interface, and computer control. The system operates referring the standard test method for calibration of helium leak detectors by use of secondary standards (ASTM F78). This reference standard leak calibrator (ref.) was calibrated near 23°C by comparison with Flowmeter of the national institute standards and technology (NIST) primary Leak Standard, using a combination of direct flow and upper to lower chamber flow division technique. The present estimate of the total uncertainty in the measured leak rate of this artifact at 23°C at the time of test is 5.2%. This includes a systematic uncertainty of 2.0% in Flowmeter of the NIST primary Leak standard and 3.2% random errors in the measured leak rate of this artifact. The errors represent three standard deviations. In order to optimize the overall accuracy in the use of this artifact, it should be stored with any shutoff valve open (a dust cover can be used to protect the vacuum port). The leak artifact should be stored at the temperature at which it will be used for at least 24 hours and pumped for least 3 hours prior to use. Sufficient time should also be allowed for the system on which the leak artifact is to be used to come to equilibrium with the environment. The present research has demonstrated the high stability of the calibration system for helium leak calibrator, and its capabilities of conducting calibration for helium leak calibrator with great efficacy.

VT-TuP6 Status of the FRIB Vacuum System Design, P. Gibson, B. Durickovic, P. Guetschow, FRIB, R. Kersevan, CERN, M. Leitner, D. Leitner, L. Lingy, F. Marti, G. Morgan, FRIB, D. Sanderson, NSCL, M. Schein, M. Shuptar, FRIB

Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, MI 48824 USA

The Facility for Rare Isotope Beams (FRIB), a new national user facility for nuclear science funded by the U.S. Department of Energy - Office of Science and operated by Michigan State University (MSU), is currently being designed and established to provide intense beams of rare isotopes. It will enable scientists to make discoveries about the properties of these rare isotopes in order to better understand the physics of nuclei, nuclear astrophysics, fundamental interactions, and applications for society. The FRIB driver linac accelerates ions as heavy as 238U to energies beyond 200 MeV/u at beam powers up to 400 kW. Machine maintainability requires an average uncontrolled beam loss below 1 W/m. Baseline vacuum levels have been established to support this requirement and pumping systems have been defined. Vacuum levels in high loss regions such as beam stripping and collimation areas have also been evaluated and pumping systems defined. Value engineering has been applied to minimize the number of different vacuum pump and beam box types and sizes where possible. The pumpdown and operating vacuum of large, complex Target System chambers has been analyzed and pumping systems defined. This talk reports on the status of the FRIB vacuum system design.

This material is based upon work supported by the U.S. Department of Energy Office of Science under Cooperative Agreement DE-SC0000661

VT-TuP7 PLS-II Vacuum System Commissioning, C.D. Park, S. Chung, T. Ha, C.K. Kim, M.S. Hong, H.C. Kwon, Y.D. Joo, Pohang University of Science and Technology, Republic of Korea

è Pohang Light Source's upgraded machine (PLS-II) has been on commissioning stage since the installation completed in June 2011. PLS-II storage ring vacuum system is designed to maintain a base pressure in the low 10^{-9} Torr region for the beam-gas scattering lifetime in excess of 20 hours, and the vacuum components, especially photon absorbers, are designed to endure 3 GeV, 400 mA beam operation. Several issues regarding the vacuum system will be presented, such as BPM stability from mechanical or electrical point of view, vacuum chamber displacement, photon absorbers, and vacuum related events during the commissioning.

* VT Student-Built Vacuum Systems Poster Competition

VT-TuP8 An Application of Electron Stimulated Desorption Technique to Measure the Isotherm and the Mean Residence Time of Hydrogen Physisorbed on a Metal Surface, H. Shimizu*, N. Inose, T. Miura, I. Arakawa, Gakushuin University, Japan

Physorption characteristics of hydrogen on a cold metal surface were investigated in the range of a monolayer for the purpose of establishing a cryopumping technique in extremely high vacuum. Electron stimulated desorption (ESD) technique was applied to measure the adsorption density of hydrogen molecules. Hydrogen was adsorbed on a copper substrate at 3 - 4.2 K of temperature and was irradiated with a pulsed electron beam of a few hundreds nsec pulse width and 45 - 170 eV electron energy. ESD ions, H⁺, H₂⁺, and H₃⁺, were mass-separated by a time-of-flight measurement with the acceleration bias of 100 V. In submonolayer range, the signal intensity of ESD H⁺ was a useful measure for the amount of physisorbed H₂ and the monolayer completion of H₂, which was accompanied by two dimensional phase condensation, was detected by the rapid rise of H⁺ intensity followed by its saturation.

An adsorption isotherm was obtained as the ESD signal intensities of H⁺ as a function of the hydrogen pressure, which was gradually increased from 10⁻⁹ to 10⁻⁴ Pa so as to keep adsorption equilibrium. The observable lower end of the isotherm was 1x10⁻⁸ Pa of the equilibrium pressure and 0.001 of H₂ coverage, which was limited by back ground signal of H⁺ originated from water or hydrocarbons adsorbed on the metal substrate. Though the validity of the assumption that the H⁺ desorption yields is proportional to the H₂ physisorption density should be further examined, the H⁺ yield curve between 1x10⁻⁸ and 2x10⁻⁶ Pa represents the adsorption isotherm between 1 and 0.001 of H₂ coverage.

Two transient methods were applied to measure the mean residence time, τ , of physisorbed H₂. A time development of ESD H⁺ intensity was monitored after either (1) a stepwise H₂ pressure rise to a certain fixed value or (2) YAG laser cleaning of the substrate under a constant H₂ pressure. In either case, the time constant of the increase of ESD H⁺ intensity is the direct measure of the mean residence time of H₂. Typical value of τ obtained by the method (1) at nearly monolayer and at 4.2 K was 62±3 sec, which corresponds to 1.19 kJ/mol of the adsorption energy on assumption of $\tau_0 = 10$ -13 sec.

The adsorption isotherms and the close examinations of the dependency of τ on the temperature and the coverage can reveal the fundamental properties of the hydrogen physisorption in low pressure and low coverage range.

VT-TuP9 Radiative Heating from a Magnetically Levitated Turbo Pump, H. Bull†, CNSE, A. Antohe, SEMATECH, G. Kane, G. Denbeaux, CNSE

Thermal management within vacuum chambers is sometimes a concern for temperature dependent vacuum processes. We describe a phenomenon in which using a magnetically levitated turbo molecular pump causes internal heating of a chamber. We measured the heat load based on a thermocouple inside the vacuum chamber, exposed line of sight to the magnetically levitated turbo pump. The measurements showed a temperature change of more than 10C based on the heat load from the pump. We also measured the effect of any heating due to gas flows and found that the dominant effect was not related to gas flows and was likely simply radiative heat transfer from the pump. The source of the heating of the pump is likely Ohmic heating due to eddy currents in the turbo pump.

VT-TuP10 Novel Light Sputter Ion Pump with Neodymium Iron Boron Magnets and the Low Outgassing Body, T. Ha, S. Chung, C.D. Park, Pohang University of Science and Technology, Republic of Korea

A light sputter ion pump for ultra high vacuum was fabricated using 5-mm-thick neodymium iron boron (NdFeB) magnets. The weight of the pump was reduced by 30%, compared with conventional sputter ion pumps using ferrite magnets. For the sake of uniformity of magnetic field, three pieces of magnet were combined into a 100 mm x 150 mm magnet block for the 30 l/s pumping element. The authors coped with the low working temperature (below 150oC) of the NdFeB magnets by forming a pure and dense chromium oxide film on the inner surface of the stainless steel pump body. With this vacuum thermal oxidation, the outgassing rate of water and hydrogen was considerably reduced so that a high temperature bakeout was unnecessary. The ultimate pressure achieved by the developed pump was much lower than 10⁻¹⁰ mbar even with a low bakeout temperature at 100oC.

VT-TuP11 Expanded Capability of Measuring Pumping Speed of Dry Vacuum Pumps Using Calibrated Sonic Nozzles, W.S. Cheung, S.H. Nam, W.J. Kim, J.Y. Lim, KRISS, Korea

This paper addresses technical issues in expanding the capability of measuring the pumping speed of dry vacuum pumps using a built-up block of multiple calibrated sonic nozzles. The first challenging issue comes from the technical limit that their calibration results available from the flow measurement standard laboratories do not fully cover the low vacuum measurement range of 10 ~ 1000 mbar although the use of sonic nozzles for precision measurement of gas flow has been well established in national metrology institutes. In order to tackle the first technical issue, the constant volume flow measurement method dedicated for the vacuum range of 10 ~ 1000 mbar is exploited to calibrate the discharge coefficient multiplied by the cross-section area of each sonic nozzle used in the built-up block of sonic nozzles. On the other hand, the discharge coefficient multiplied by the cross-section area of each sonic nozzle in the upstream pressure range of 2 ~ 10 bar is calibrated in the gas flow standard laboratory. These combined calibration methods are illustrated to enable each sonic nozzle to cover the three decade measurable range 10 mbar ~ 10 bar. The second technical issue comes from a logical way of stacking multiple sonic nozzles to expand the capability of measuring the pumping speed of dry vacuum pumps. A target value of the upper limit of pumping speed in this work was selected to be 4,500 m³/h as the Korean flat display manufacturers requested to KRISS. On the onset of this work, sonic nozzles of 5 mm throat diameter were machined to fulfill the upper limit pumping speed by stacking three or four nozzles in the built-up block. Of course, small sized nozzles (0.1, 0.16, 0.5, and 1.0 mm throat diameter) were also machined in this work to cover the low and mid range of the throughput of 0.1 ~ 100 mbar-l/s. A prototype of the built-up block with the capacity of installing six sonic nozzles simultaneously is under test. Its design throughput was to be about 125,000 mbar-l/s. This prototype is expected to provide a possible 'on-line pumping speed tester' with the capability of 4,500 m³/h or more, applicable not only to the on-site flat display processes but also to the semiconductor processes.

VT-TuP12 Study on Improvement of Predictive Maintenance of Dry Vacuum Pumps Using an Adaptive Parametric Model of State Variables, S.H. Nam†, W.J. Kim, J.Y. Lim, W.S. Cheung, KRISS, Korea

This paper introduces unique statistical features extracted from the measured state variables of dry vacuum pumps in the semiconductor processes. They were found to have three distinctive means and overlapped distributions, not a single normal distribution. More specifically, two distinctive distributions near the upper and lower asymptotic bounds are obviously observed from the gas-loaded states of the vacuum pump and the third one from the idle states. These observations have provided new motivations of not only separating the pump operation state into the gas-loaded and idle states but also modeling the upper and lower bounds as a separated distribution. A linear adaptive parametric model (APM) is proposed such that their linear trends of each state variable are shown to be mapped onto their model parameters. Those estimated model parameters are used to construct the batch data obtained after each process. The APM-based batches are also exploited to construct the batches under the normal operating conditions (NOC) such that the major eigenvectors of the NOC batches are used to diagnose the current process batch data. It should be noted that the APM-based batch provides a dramatic reduction of memory usage and computation time (for example, 1~2 % memory usage and 10 times faster computation time) in comparison to the conventional dynamic-time wrapping methods. The feasibility of the proposed APM for the predictive maintenance of dry vacuum pumps is demonstrated to be successful by illustrating test results obtained from the six dry vacuum pumps.

This paper proposes the use of two statistics, the Hostelling's T² and the sum of squared residuals, in order to improve the reliability of the predictive maintenance and self-diagnostics of vacuum pumps. The first one is exploited to examine what amount of similarity the current process batch has in reference to the normal operation conditions and, furthermore, the second is to examine what contribution the current process batch provides to the noise space. The two proposed statistics are examined to quantitatively analyze the reliability and improvement of the predictive maintenance and self-diagnostics developed in this work.

VT-TuP13 Modeling of Energy Consumption Characteristics of Low Vacuum Dry Pumps, W.J. Kim†, S.H. Nam, W.S. Cheung, KRISS, Republic of Korea, M.K. Ko, Konyang University, Republic of Korea, J.Y. Lim, KRISS, Republic of Korea

This presentation addresses measurements related to energy, utilities and materials usage on dry vacuum pumps. A particular course of action was taken into account with regard to the measurement into equivalent energy suggested by SEMI Document 4399.

* VT Student-Built Vacuum Systems Poster Competition

Recent SEMATECH and SEMI studies showed that 50 ~ 60 % of equipment power is used for vacuum pumps. Currently vacuum pump suppliers have responded by reducing power consumption and cooling water flow requirement in energy consumption at the component level. Actual process studies showed that for some processes, the energy consumption level did not change significantly during idle and processing operation modes. However, specified studies in experimental scale to characterize the energy consumption pattern have not been reported yet.

We have performed a simulation study to characterize energy consumption pattern in the idle and process modes.

The pressure range of about 0.1 to 50 mbar for 7 minutes was assigned to the simulated process mode, meanwhile the pressure of <0.1 mbar for 3 minutes to the idle mode. The integrated characteristics evaluation system for dry vacuum pumps has been utilized to gather the dry pump characteristics data for the simulation. Roots, claw, classical screw, and multi-stage type vacuum pumps supplied from the manufacturers have been evaluated using the evaluation system in terms of ultimate pressure, pumping speed, power consumption, vibration, sound power as well as nitrogen purge, cooling water rate from the single pump monitoring system in time-synchronized mode. This study includes the application of the SEMI S23-0705 standards – A Guide for Conservation of Energy, Utilities and Materials Used by Semiconductor Manufacturing Equipment.

The estimated power consumption per pump per year was ranged from 10 to 30 MWh and 15 to 50 MWh for 600 ~ 1200 m³/h dry pumps in idle and processing modes, respectively. The utility energy consumption was also ranged from 5 to 10 MWh and 10 to 30 MWh, respectively. More specific energy consumption patterns with respect to the pressure are also presented.

In this work we suggest that the correlation mechanism dependant on the actual process lines should be carefully analyzed and furthermore understood, for example, the relationship between cooling water flow rate and temperature variation during processes. Simple characteristic modeling of energy consumption patterns dependent on dry and roots pump types are also discussed.

Acknowledgements: Results are partially attributed to two national projects (Contract Nos. 10031858 and 10031836) sponsored by the Korean Ministry of Knowledge Economy.

VT-TuP14 Dry Vacuum Pump, J.Y. Lim, W.J. Kim, S.H. Nam, KRISS, Republic of Korea, S.Y. In, Korea Atomic Energy Research Institute, Republic of Korea, D.Y. Koh, Korea Institute of Machinery and Materials, Republic of Korea, W.S. Cheung, KRISS, Republic of Korea

Advanced industrial processes such as semiconductor and display manufacturing continuously requires the precise measurement and control of the low mass flow of gases. The requirements include very low mass flows of less than 1scm or 2×10^{-2} mbar·l/s. However, the lower limit of traceable mass flow ranges are not well defined even in the sophisticated NMIs (National Measurement Institute). Since the primary standard for mass flow (kg/s) must provide a method for deriving mass flow directly from its base units, almost all mass flow systems heavily rely on the gravimetric method. Current technical measurement limit of mass flow rate with the gravimetric method is 2×10^{-8} kg/s ($10^{-2} \sim 10^{-3}$ mbar·l/s).

Ensuring mass flow traceability ability in the range of greater than 10^2 mbar · l/s with the Korea Research Institute of Standards and Science (KRISS) standard system, an attempt to trace the lower mass flows down to 10^{-4} mbar · l/s has been undertaken with constant volume flow meters (CVFM), sonic nozzle systems, and orifice method. In this work we briefly demonstrate the systematic attempt for the completion of the traceability chain from 10^4 to 10^{-4} mbar · l/s.

Meanwhile the most effective way of ensuring completion of the traceability chain is, to say, characteristics monitoring of the mechanical rotary machines such as vacuum pumps used in advanced industry since these pumps have normally clean and smooth pumping capabilities during their stable operation.

Utilizing the combined mass flow systems mentioned above, the measurement of pumping speed has been performed with the throughput and orifice methods dependent on the mass flow regions. However, in the HV range of the molecular flow region, the high uncertainties of the gauges, mass flow rates, and conductance are too critical to precisely accumulate reliable data. In order to solve the uncertainty problems of pumping speeds in the HV range, we introduced an SRG with 1 % accuracy and CVFMs to measure the finite mass flow rates down to 10^{-3} mbar · l/s with 3 % uncertainty for the throughput method. In this way we have performed the measurement of pumping speed down to less than 10^{-6} mbar with an uncertainty of 6 %. In this article we suggest that the CVFM has an ability to measure the conductance of the orifice experimentally with flowing the known mass through the orifice chambers, so that we may overcome the discontinuity problem encountering during introducing two measurement methods in one pumping speed evaluation sequence.

VT-TuP16 An Evaluation of the Outgassing Rates of Stainless Steel Vacuum Chambers Subjected to Different Heat Treatments and Coatings, M.A. Mamun*, Old Dominion University, P.A. Adderley, M.L. Stutzman, M. Poelker, Thomas Jefferson National Accelerator Facility

We present the results of an extensive study of the outgassing rates of four nominally identical stainless steel vacuum chambers. Chambers were either heat treated (semi-vacuum) at 400°C, coated with TiN, coated with SiO₂ (SilcoGuard™), or heat treated then coated with SiO₂. The outgassing rates were measured at a variety of room temperatures to determine the temperature dependence and compare to theoretical models of diffusion- or recombination-limited outgassing. In addition, outgassing rates were evaluated following venting to air and re-baking at 150°C and 250°C to simulate the impact of putting a chamber into service.

VT-TuP17 A Quantitate Examination of Venting Trapped Volumes Due to Fasteners, C. Bryson, Apparati Inc.

Why we care

The need addressed here is the problem of pumping out internal volumes in vacuum systems that may be blocked by construction methods. Vented screws are a common approach to reducing this problem. This discussion will focus on evaluating the magnitude of the problem and various solutions. Calculations on conductance for vented and unvented screws and some accessories will be given. These conductance values will be used to model different common configurations that have different amounts of trapped volumes.

The conclusions are clear; vented screws and venting accessories are essential.

Summary

The direct measurements on un-vented screws were flawed because the screws tended to seal to varying degrees by the head of the screw. A use of a vented washer helped but the leak rate was hard to measure because of variability. Direct comparison to calculations is difficult because the tolerances in screw fasteners. There are large differences in conductance given the dimensions of the clearance between the screw and the mating fixtures. Calculating the ranges of conductance that can be expected for unvented screws, venting channels and internal volumes were done for different applications in vacuum systems. Ranges in excess of a factor >15 were encountered for a single division of a particular fastener. Some comparisons with data shed insight to the nature of the task.

The result is a "set of ground rules for design" based on the quantitative information derived. Examples are given for different designs with different parameters in terms of vacuum performance.

* VT Student-Built Vacuum Systems Poster Competition

Wednesday Morning, October 31, 2012

Vacuum Technology

Room: 14 - Session VT+AS+SS-WeM

Surface Analysis and Vacuum Manufacturing for Accelerators

Moderator: M.L. Stutzman, Thomas Jefferson National Accelerator Facility

8:00am **VT+AS+SS-WeM1 Manufacturing and Welding Processes for TPS Large Aluminum Bending-Chambers and 14 m Vacuum Cells.** *C.L. Chen, C.C. Chang, C.K. Chan, Y.C. Yang, T.Y. Lee, G.Y. Hsiung, J.R. Chen*, NSRRC, Taiwan, Republic of China

A unique manufacturing and welding technique has been developed for building the 3 GeV Taiwan Photon Source (TPS) large aluminum bending chambers and 14-meter vacuum cells. There are total 48 bending chambers, which are about 3.8 meters long each. Combined with an appropriate manufacturing processes, such as with a precise CNC machine, lubrication with pure alcohol and cleaning with ozonated water, the aluminum chambers have an oil-free interior surface finished for an ultra-high vacuum environment before aluminum welding. Ozonated water has a high oxidation potential and can remove most organic contaminations. It is used to effectively clean aluminum chambers' surfaces, and provides with the lowest outgassing yield. After the bending chambers are cleaned with ozonated water, the chambers are moved to a welding room for following welding processes. A novel automatic gas-tungsten arc-welding (GTAW) system has been established at NSRRC for welding the aluminum bending chambers. This welding system has a XY stage that is built and configured to provide high-performance positioning along multiple welding axes. The automatic welding system comprises six welding torches to implement simultaneously two longitudinal side welds of a bending chamber, and is innovative in using computer-based software to control the welding movements and the welding parameters of the six-torch output. In traditional manual welding, the key success factors focus on elimination as much as possible the distortions of structural assemblies. The six-torch welding and a clamp-free approach together address the issue of reducing distortion and minimizing residual stresses with a novel one-step welding process. In addition, on-site welding sequence is introduced for assembling two straight and two bending chambers into one 14-meter vacuum cell. From the beginning of CNC machining to the end of vacuum cell assembly, deformations through all process sequences are measured and controlled under 300 μm . In this paper, both the manufacturing sequences, vacuum data and statistical analysis of deformation control are presented in detail.

8:20am **VT+AS+SS-WeM2 A High Power Electron Beam Stop for Cornell ERL Prototype Injector.** *X. Liu, Y. Li, K.W. Smolenski, I. Bazarov, B.M. Dunham*, Cornell University

The electron beam stop for Cornell University's Energy Recovery Linac (ERL) prototype injector was designed and manufactured for 600 kW electron beam power at beam energies between 5 and 15 MeV. To minimize neutron production from high energy electrons, aluminum was chosen over copper for the construction material. It consists of a 20 mm thick main body with machined outer cooling channels and a tight fit jacket, with the thickness mainly determined by the stopping power of the material. The stop body also serves as the vacuum envelope. The stop body is made of three sections, which are electron-beam welded together. It has a cylindrical shape with a cone at the end, about 0.5 m in diameter and 3 m in overall length. Flexibility is allowed at the body-jacket joint to minimize the thermal stress. The naturally small ERL electron beam is enlarged and rastered in a circular pattern using magnets at the entrance. The enlarged electron beam strikes the stop surface at an average angle of about 8 degrees. The electron scattering inside the stop body was simulated using GEANT4, and the inside profile of the body was optimized so that the thermal load is the most evenly distributed over the whole body. A quadrant detector is equipped at the entrance of the stop to monitor the electron beam centering and rastering. An array of thermocouples is installed on the outside surface of the jacket, providing a rough map of the heat load distribution. Gases generated in the close-circuit cooling water by radiolysis are vented and the concentration of hydrogen is monitored. The stop has been in operation since October 2008, and has been tested up to 250 kW to date.

8:40am **VT+AS+SS-WeM3 Ion Pump Starting Behaviour at High Pressures - Influence of Pump Design Diode / Triode and Power Supply.** *M. Thierley, C. Paolini*, Agilent Technologies, Italy

Today ion pumps are broadly seen as pumps for good high vacuum and ultra-high vacuum environments. Operated at these low pressures, the power consumption of ion pumps is also very low, making ion pumps one of the most energy efficient vacuum pump technologies. Power supplies, however, with several hundred Watts of power continue to be used, as in the past decades, often based on the fear of not being able to start the ion pump; historic issues associated with higher pressures. In this presentation/paper, the differences in starting behaviour of Diode and Triode pump elements are discussed, based on experimental data. Questions addressed will include; how does the pumping speed of these elements change while starting with voltage and current? What impact does the power and design of the pump control unit have on the start and the pump down time of the vacuum system? What is the impact of the power supply unit's technology (ie. classic transformer based design vs. more modern switching power supplies)? In addition to controller experimental data and discussion of the operation theory of the pump elements, pictures of the actual plasma development inside the pump will be featured.

9:00am **VT+AS+SS-WeM4 Superconducting Niobium for Accelerator Cavities: Status and Prospects.** *M.J. Kelley*, Jefferson Lab and College of William & Mary **INVITED**

Radiofrequency accelerator cavities of superconducting niobium are the technology of choice for a number of recent and coming particle accelerators, largely because of their cost-for-performance. The principal aspects of performance are the amount accelerator needed to achieve a required final beam energy (accelerating gradient, E_{acc}) and energy consumption (cavity quality factor, Q_0). The former impacts chiefly initial cost; the latter both initial and operating costs. Research and development efforts are bearing fruit for both. Gaining the benefits need not await the construction of new accelerators or major upgrades, as accelerator modules are regularly cycled out of existing machines. A challenge that is under-appreciated by physics researchers, but is well familiar to the AVS community, is the manufacturing excellence needed to translate research progress into hardware on the ground.

Authored by Jefferson Science Associates LLC under US DOE Contract De-AC05-06OR23177

9:40am **VT+AS+SS-WeM6 Niobium Nitride Thin Films and Multilayers for Superconducting Radio Frequency Cavities.** *W.M. Roach*, D.B. Beringer, Z. Li*, The College of William and Mary, *J.R. Skuza*, National Institute of Aerospace, *C. Clavero, R.A. Lukaszew*, The College of William and Mary

Niobium nitride in thin film form has been considered for a number of applications including multilayered coatings onto superconducting radio frequency (SRF) cavities which have been proposed to overcome the fundamental accelerating gradient limit of 50 MV/m in niobium based accelerators [1]. In order to fulfill the latter application, the selected superconductor's lower critical field, H_{c1} , must be larger than that of niobium and separated from the niobium surface by an insulating layer in order to shield the niobium cavity from field penetration, therefore allowing higher field gradients. Thus, for the successful implementation of such a multilayered stack it is important to consider not just the material's inherent properties, but also how these properties may be affected in thin film geometry and also by the specific deposition techniques used. Here, we present the results of our correlated study of structure and superconducting properties in niobium nitride thin films. Additionally, we explore how growth parameters can affect the surface morphology, since the quality of the surface has major implications on the ultimate performance of SRF cavities. Combining our findings on the surface morphology, microstructure, and superconducting properties in niobium nitride thin films, we discuss their potential application in multilayered coatings for accelerator cavities.

[1] A. Gurevich, Appl. Phys. Lett. **88**, 012511 (2006).

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* ASSD Student Award Finalist

10:40am **VT+AS+SS-WeM9 Characterization of Anisotropic Surface Morphology in Epitaxial Superconducting Thin Films by Wavelet Analysis.** *D.B. Beringer, J.B. Hackett, W.M. Roach, R.A. Lukaszew*, The College of William and Mary

Surface morphology and interface roughness are critical factors impacting the ultimate performance of many thin film materials and nano-scale devices. Next generation superconducting radio frequency (SRF) materials for particle accelerator cavities depend upon the ability to tailor and finely control the microstructure and morphology of superconducting / insulating /superconducting (SIS) multilayer thin film structures. The evolving surface of grown epitaxial thin films, influenced by nucleation and growth kinetics, may exhibit dendritic or fractal patterning where the resulting anisotropic features dominate a coarsening morphology. As such, a quantitative understanding of superconducting thin film morphology and the thin film deposition parameters leading to optimal SRF surfaces is desirable. Quantitative characterization of surface morphology is typically achieved with Fourier transform (FT) analysis and fractal characterization; however, this approach suffers intrinsic limitations as the FT is localized in the frequency domain and therefore cannot differentiate between specific features with isolated spatial coordinates. Wavelet analysis transcends these limitations by effectively isolating and quantifying surface features belonging to a designated length scale, thus enabling independent analysis of local surface features with varied spatial resolutions. Here we present our work with surface characterization by wavelet analysis of epitaxial superconducting Nb thin films.

11:00am **VT+AS+SS-WeM10 NbN-AlN-Nb Multilayer Thin Films for Superconducting Radio Frequency Cavities.** *Z. Li, W.M. Roach, D.B. Beringer, C. Clavero, R.A. Lukaszew*, College of William and Mary

Linear accelerators that are used in high-energy or nuclear physics experiments use superconducting radio frequency (SRF) cavities made with bulk Nb. However, as technology is improved for bulk Nb cavities, the accelerating gradient for these cavities is reaching the fundamental limit of 50 MV/m. Since the critical surface of Nb in SRF cavities is less than one micron, it is possible to use thin films and multilayers to overcome the accelerating gradient limit. It has been proposed to apply a superconductor-insulator-superconductor (SIS) multi-layer structure onto Nb based cavities in order to provide an improved lower critical field (H_{c1}) that will shield the Nb and therefore allow for an increase in the accelerating gradient [1]. NbN is one of the superconductors that may be implemented in this SIS structure. However, the choice of insulator is crucial in determining the performance of NbN thin films. Here, we present our study of epitaxial thin films prepared on both MgO and AlN templates. The effect of substrate choice on microstructure and superconducting properties is explored in order to determine which insulator provides optimal performance of NbN thin films for SRF applications.

[1] A. Gurevich, *Appl. Phys. Lett.* **88**, 012511 (2006).

Friday Morning, November 2, 2012

Applied Surface Science

Room: 20 - Session AS+TF+VT-FrM

Surface Analysis using Synchrotron Techniques

Moderator: A. Herrera-Gomez, UAM-Azcapotzalco and CINVESTAV-Queretaro, Mexico, J.C. Woicik, National Institute of Standards and Technology

8:20am **AS+TF+VT-FrM1 Surface and Interface Analyses by X-ray Absorption and Hard X-ray Photoemission Spectroscopies**, *Q. Xiao, X. Cui*, Canadian Light Source, Canada, *H. Piao*, General Electric Global Research Center, *Y.F. Hu*, Canadian Light Source, Canada, *T.K. Sham*, The University of Western Ontario, Canada

Synchrotron-based techniques, such as X-ray absorption spectroscopy (XAS) and variable energy X-ray photoemission spectroscopy (XPS) are increasingly applied to the characterization of surfaces and interfaces of advanced materials. This presentation will introduce the XAS and variable energy XPS capabilities in the study of thin films and nanomaterials at the Canadian Light Source—the third generation synchrotron in Canada. Advantages of these techniques over the conventional techniques (such as lab-based XPS) will be demonstrated using examples in studies of two types of materials: (1) Gate oxide development on SiC and (2) heterogeneous nanocatalysts. In particular, examples using the recently commissioned high energy XPS at the SXRMB beamline (up to 10 KeV) will be highlighted.

8:40am **AS+TF+VT-FrM2 Differences in the Electronic Structure Highly-Oriented Films of H₂, Fe-, Co-, and Cu-Phthalocyanines Revealed by NEXAFS Spectroscopy**, *T.M. Willey, M. Bagge-Hansen, J.R.I. Lee, R. Call, L. Landt, T. van Buuren*, Lawrence Livermore National Laboratory, *C. Colesniuc, C.M. Monton, I. Schuller*, University of California, San Diego

Phthalocyanines are extensively studied as molecular semiconductor materials for chemical sensors, dye-sensitized solar cells, and other applications. Phthalocyanines offer high tunability through the choice of metal center atom; nearly all transition metals and many other heavier elements can reside at the relatively stable square planar center of the phthalocyanines. H₂, Fe-, Co-, and Cu-phthalocyanine molecules in films deposited on gold substrates show prostrate orientation, as opposed sapphire substrates, where phthalocyanines stand in a more upright conformation under deposition conditions used. Angular dependence in NEXAFS, commonly attributable to π^* and σ^* resonances, in both carbon and nitrogen K-edges, quantify the orientational order. H₂-phthalocyanine shows the cleanest angular dependence, with nearly no intensity in the π^* regime with normal beam incidence. Metal L-edges in prostrate films, on the other hand, have dramatic variation in angular dependence of resonances into empty states. Fe- and Co- resemble the K-edges; StoBe DFT shows that the lowest-energy allowed resonances are indeed molecular π^* states, with a high degree of mixing with the d_{xz} and d_{yz} orbitals of the metals. In contrast, the intense, in-plane resonance of the Cu-PC L-edge LUMO resembles a molecular σ^* state. Confirmed by StoBe, the $d_{x^2-y^2}$ character at the Cu center is responsible for this intense in-plane resonance. NEXAFS thus directly probes the electronic structure, illuminating the uniqueness of Cu-compared to H₂, Fe-, and Co- phthalocyanines.

9:00am **AS+TF+VT-FrM3 Hard X-ray Photoelectron Study of Graphene/ h-BN Layer Structures Grown on Polycrystalline Cu Substrates**, *L. Kövér*, MTA ATOMKI, Hungary, *L. Tapasztó*, Inst. Tech. Physics and Materials Sci. & Korea-Hungary Joint Lab for Nanosciences, Hungary, *C. Hwang*, KRISS & Korea-Hungary Joint Lab for Nanosciences, Republic of Korea, *L.P. Biró*, Inst. Tech. Physics and Materials Sci. & Korea-Hungary Joint Lab for Nanosciences, Hungary, *I. Cserny, J. Tóth, A. Csik*, MTA ATOMKI, Hungary, *W. Drube, S. Thiess*, Deutsches Elektronen-Synchrotron DESY, Germany

INVITED

Graphene-hexagonal BN layer structures are recently in the focus of interest having a great potential importance as promising candidates to be utilized in many electronic and spintronic ultrathin device applications. Hard X-ray photoelectron spectroscopy (HAXPES) based on application of synchrotron radiation for excitation is a useful tool for revealing multilayer structures nondestructively providing information on the chemical state of the components at the surface and (even in the case of deeply embedded) interface layers. The aim of the present study is to test the applicability of the HAXPES method for characterizing graphene-hexagonal BN layer systems. h-BN and single layer graphene/multilayer h-BN structures were grown on polycrystalline Cu substrates using the Chemical Vapor Deposition technique [1]. HAXPES measurements were performed at the

BW2 beamline of the DORIS III synchrotron at DESY using the Tunable High Energy XPS facility [2] and monochromatic photons of 3000 eV energy. The surface layer structure of the same samples were also studied with conventional XPS in ATOMKI using non-monochromated Al K α radiation [3]. For obtaining information on the order and relative depth of the particular layers the dependence of the XPS and HAXPES spectra on the angle of emission of photoelectrons was also studied. Our results demonstrate the advantage of the combination of XPS and HAXPES measurements, the use of the Auger parameter for identifying the chemical state of the components at the interface between the layer structure and the substrate, the removal of the overlap between the Auger spectra from the substrate and the photoelectron spectra of the overlayer structure in the HAXPES spectra. In addition, HAXPES, due to the high energy resolution and sensitivity applied, makes possible the separation and identification of the contributions from atoms in different chemical states to photoelectron peaks of the components of the layer structure, and the quantitative estimation of their relative intensity, even at near grazing photon beam incidence (high surface sensitivity).

The research leading to these results has received funding from the European Community's Seventh Framework Programme (FP7/2007-2013) under grant agreement n° 312284, by OTKA grants PD 84244, K 101599 and in the framework of the Korea- Hungary Joint Laboratory for Nanosciences.

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9:40am **AS+TF+VT-FrM5 Beyond Hard X-ray Photoelectron Spectroscopy: Simultaneous Combination with X-ray Diffraction**, *G.R. Castro, J. Rubio-Zuazo*, SpLine at the European Synchrotron Radiation Facility, France

Nowadays, the great challenge in materials science is the incorporation of complex systems in the area of the nano-technologies. A fundamental aspect is the production of materials with specific and controlled properties. Many of these materials are aggregates of different components, frequently multilayer thin films where the interface and the surface play a key role. Therefore, it is very important to develop an experimental set-up capable to investigate different aspects under identical experimental conditions, in particular to differentiate between surface and bulk properties.

*Hard X-ray photoelectron spectroscopy (HAXPES) is a powerful novel emerging technique for bulk compositional, chemical and electronic properties determination in a non-destructive way. It benefits from the exceptionally large escape depth of high kinetic energy photoelectrons enabling the study of bulk and buried interfaces up to several tens of nanometres depth. At SpLine, the Spanish CRG beamline at the European Synchrotron Radiation Facility (ESRF), we have developed a novel and exceptional set-up that combine HAXPES and X-ray diffraction (X-ray Reflectivity, Surface X-ray Diffraction, Grazing Incidence X-ray Diffraction and reciprocal space maps). Both techniques can be operated simultaneously on the same sample and using the same excitation source. The set-up includes a robust 2S+3D diffractometer with its main axis vertical hosting an UHV chamber equipped with a unique photoelectron spectrometer (few eV < E_{kin} < 15keV), X-ray tube (Mg/Ti), 15 keV electron gun and auxiliary standard surface facilities: MBE, ion gun, LEED, sample heating/cooling system, leak valves, load-lock port, etc.. The photon energy ranges between 7 and 45 keV. The HAXPES analyzer is an electrostatic cylinder-sector (FOCUS HV CSA), with a compact geometry and high transmission due to second order focusing. The analyzer is capable to handle kinetic energies both up to 15 keV and down to a few eV with the same analyzer setup and power supply. The SpLine station offers a unique opportunity to obtain, on a same sample and under identical experimental conditions, simultaneous information about the electronic properties, chemical composition and geometric/crystalline structure of bulk, buried interfaces and surfaces. This *novel tool* for non-destructive characterization of bulk and buried interfaces is available to the *scientific community*.*

In this contribution, we will present a general view of HAXPES-XRD station available at SpLine. Three aspects will be specially addressed: physical background, experimental set-up and selected examples.

10:00am **AS+TF+VT-FrM6 Spectroscopic Imaging using Vector Potential Photoelectron Microscopy**, *R. Browning*, R. Browning Consultants

A new class of electron microscope, vector potential photoelectron microscopy (VPPEM) has been developed. This microscope will enable the chemical microanalysis of a wide range of samples using photoelectron spectroscopy (PES). The microscope is a full field spectroscopic imaging technique with a very large equivalent depth of focus. The unique imaging properties of this method opens up many experimental opportunities including the chemical microanalysis of a wide range of real world samples. Highly structured, three dimensional samples, such as fiber mats and fracture surfaces can be imaged, as well as insulators, and magnetic materials. The new microscope uses the vector potential field from a solenoid magnet as a spatial reference for imaging. A prototype instrument has demonstrated imaging of Au grids, uncoated silk, magnetic steel wool, and micron sized single strand tungsten wires.

10:20am **AS+TF+VT-FrM7 Trends in Synchrotron-based Photoemission; High Energy and High Pressure**, *H.J. Bergersen, J. Åhlund, R. Moberg*, VG Scienta, Sweden

The fields of Hard X-ray Photoelectron Spectroscopy (HAXPES) and High Pressure Photoemission (HiPP) are growing fast. In this contribution we present instrument development and results within HAXPES and HiPP as well as the merged field of HiPP-HAXPES.

Photoelectron spectroscopy (PES) is an excellent tool in surface science due to the possibility to probe electronic and geometric structure. During the past decade Angle Resolved Photoelectron Spectroscopy (ARPES) has had a remarkable upswing, due to the development of parallel angular detector analyzers, and is today used routinely for band mapping, depth profiling and X-ray diffraction (XPD) in the Ultra Violet (UV) and soft X-ray regime. With higher energies (hard X-rays), in combination with improvements in PES detection techniques, this tool can be extended to the HAXPES regime, enabling studies of bulk materials. Here we demonstrate new development of analysers capable of measuring angular resolved spectra in the High Energy regime as well as results obtained using such analyzers.

Experiments done under normal surface science conditions (Ultra High Vacuum) are of limited use in some applications, e.g catalysis, due to the pressure gap problem. This motivates the study of systems at ambient pressures. Here we present a HiPP instrument developed in collaboration with Advanced Light Source (ALS). This instrument allows standard PES measurements as well as spatial and angle resolved spectra at HiPP conditions. Some recent results include spatially resolved investigations of solid oxide electrochemical cells (SOC:s) and electrochemical properties of junctions.

Finally, we report on recent advances in constructing a new generation of instrumentation combining HiPP and HAXPES. A novel electron analyser, designed for optimal transmission in combination with very efficient differential pumping, will be presented together with preliminary results.

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