

## Vacuum Technology

### Room 104C - Session VT-MoM

#### Vacuum Measurement, Calibration, Primary and Industry Standards

**Moderators:** Yulin Li, Cornell Laboratory for Accelerator-Based Sciences and Education, Joe Becker, Kurt J. Lesker Company

#### 8:20am VT-MoM1 Industry Practice for Using Primary Leak Standards to Validate Calibration Methods, *Jason Alfrey*, VACUUM TECHNOLOGY INC INVITED

Accredited metrology labs are required to maintain traceability to a primary laboratory and periodically validate measurement methods. For calibration laboratories operating multiple systems, what is a suitable process to validate measurements and prove traceability while still being economically competitive?

Vacuum Technology, Inc (VTI) utilizes two methods for gas flow measurement of Leak Standards – both primary and comparison methods. Although these are well established methods in the industry, an accredited laboratory is always interested in proving it is linked to the “chain of metrology.” The following discussion presents the methods for both inter-laboratory and intra-laboratory testing using Primary Leak Standards calibrated by NIST.

#### 9:00am VT-MoM3 Fixed Length Optical Cavity for Photonic Realization of the Pascal, *Jay Hendricks, J. Ricker, P. Egan, J. Stone, G. Scace, G.F. Strouse*, National Institute of Standards and Technology

NIST is actively developing a new paradigm in the methodology of pressure and vacuum gauging and metrology. In a break with nearly 400 years of mercury based primary standards, NIST has developed a new standard that is based on the fundamental physics of light interacting with a gas. For the vacuum community, this represents a shift in how we think about the unit of the Pascal in that it will be directly related to the density of a gas, the temperature, the refractive index, and the Boltzmann constant. The photonic technique has now achieved important benchmarks in performance when compared to the existing primary standards based on mercury manometers: The photonic technique has a 20X smaller footprint, 100X faster sensing response time, 100X lower pressure range, and for an emerging technique has demonstrated impressive accuracy, reproducibility and hysteresis. Photonic sensing of the pascal has the potential to be further miniaturized, and has the key advantage that the light used for sensing the pressure can be transmitted over light-weight, high-speed fiber optic cables and networks. This talk will highlight the NIST efforts to replace our mercury Ultrasonic Interferometer Manometers (UIMs) with the new quantum-based, photonic technique. New data will be presented that shows that two independent Fixed Length Optical Cavities are now operating with part per million reproducibility and measurement agreement. The optical technique has now surpassed mercury manometer performance, and a new paradigm for vacuum metrology and realization of the SI unit, the pascal has begun.

#### 9:20am VT-MoM4 Analysis of a Quantum Based Refractometer to Replace Mercury Manometers as the Primary Standard for the United States, *Jacob Ricker, J. Hendricks, P. Egan, J. Stone*, NIST

NIST has developed a technique to measure pressure using the gas refractivity of nitrogen for pressures in the range of 1 Pa to 360 kPa. This range is critical to many application including altimetry, weather, process control, etc.; all of which require high accuracy calibration of vacuum gauges. Currently the highest claimed accuracy of a primary standard is the NIST mercury Ultrasonic Interferometer Manometers (UIMs) operating at an uncertainty of  $U(P_{UIM}) = [(6 \text{ mPa})^2 + (5.2 \cdot 10^{-6} \cdot P)^2]^{1/2}$ . NIST proposes replacement of these standards with an optical gas refractometer with an uncertainty of  $U(P_{OGR}) = [(2.0 \text{ mPa})^2 + (8.8 \cdot 10^{-6} \cdot P)^2]^{1/2}$ .

The optical refractometer has many benefits over the current UIMs, however we also need to show the feasibility of the fixed length refractometer as a primary standard. The two key requirements to define a primary standard are traceability of the standard back to the International System of Units (SI) and the ability to transfer the measurement/uncertainty to a high accuracy gauge or a transfer standard. The traceability and associated uncertainty will be discussed along with the derivation of the above stated uncertainty for the optical gas refractometer. Additionally, results of a calibration using the refractometer will be compared to that obtained using the NIST UIM. The capability and

limitations of both the refractometer and UIM will be discussed and will show that the refractometer outperforms the UIM and will be slated to replace the mercury standards in the near future.

#### 9:40am VT-MoM5 Creating Vacuum Standards in the UHV and XHV to Support Cold-Atom Physics and Other Cool Stuff, *James A. Fedchak, J. Scherschligt, M.S. Sefa, S. Eckel, D. Barker*, National Institute of Standards and Technology (NIST)

We are creating a program at NIST to develop new vacuum standards that cover the UHV and XHV. This addresses the needs of advanced manufacturing and research, including the semiconductor industry, accelerators, nanotechnology, and space science. NIST is also interested in developing metrological tools and other practical devices based on ultra-cold atoms, a technology pioneered at NIST that fundamentally operates in the UHV. Gravimeters, inertial sensing, and clocks are all examples of such devices, and any such device based on ultra-cold atoms will necessarily operate in UHV. To this end, we are presently developing a cold-atom vacuum standard (CAVS) to absolutely determine the vacuum level in the range of  $10^{-7}$  torr to  $10^{-12}$  torr. Our CAVS will use ultra-cold atoms to sense the absolute number density of gas molecules in the vacuum, and will be an SI traceable primary realization of UHV and XHV. In addition, we're developing the Cold-Core Technology program, which seeks to create a platform enabling the miniaturization of the CAVS and other practical cold-atom devices. An active UHV and XHV program is critical and necessary for this effort. This includes traditional activities such as producing and measuring low-outgassing rate materials and building dynamic expansion chambers for generating UHV pressures, as well as new efforts like building the CAVS. This talk will be an overview of these activities in light of creating the CAVS and other devices based on ultra-cold atom technology.

#### 10:00am VT-MoM6 Technical Challenges of the Cold Atom Vacuum Standard, *Julia Scherschligt, J.A. Fedchak, M.S. Sefa, S. Eckel, D. Barker*, National Institute of Standards and Technology (NIST)

NIST has recently launched a program combining cold-atom physics with vacuum metrology and has begun to build the Cold Atom Vacuum Standard (CAVS). Development of the cold atom vacuum standard presents a variety of technical challenges: we need a thorough understanding of the collision cross section of trapped atoms with background gas, we need to achieve excellent vacuum levels and prevent contamination of the test system with sensor alkali atoms, and we need a robust and user-friendly design if the device is ever to be practical for real-world applications. We will discuss progress that has been made in determining collision cross sections, measuring and reducing outgassing rates, as well as design considerations of the cold atom device itself.

#### 10:40am VT-MoM8 Investigation of a Novel Cold Cathode Ionization Gauge Geometry with Wide Range from High Vacuum to Atmosphere in a Single Gauge, *T.R. Swinney, C. Percy, Gerardo Alejandro Brucker*, Pressure & Vacuum Measurement Solutions, MKS Instruments, Inc.

Wide range vacuum gauges are in use now, however, all rely on multiple technology sensors with various mismatched pressure responses for various gases. An overview of the problems created by multiple technologies and mismatched pressure responses along with novel solutions will be presented. A new internal geometry of a cold cathode ionization gauge has been investigated and has produced a usable pressure signal from high vacuum to atmosphere, using only one gauge and only one physical electronics mechanism, namely a gaseous discharge. A redesigned electrode structure avoids mixing differences in gauge types and their responses to different gas types when using multiple gauge types over this full range. Careful choice of the anode-to-cathode spacing can sustain a gaseous discharge with the usual electric and magnetic fields. The various electrical signals used to display a pressure read-out will be presented and compared. This was accomplished during the investigation with available control circuitry.

#### 11:00am VT-MoM9 Advanced Manufacturing Techniques for Cold Cathode Ionization Gauges, *Clinton Percy*, Pressure & Vacuum Measurement Solutions, MKS Instruments, Inc.

Cold cathode ionization gauges (CCIGs) have been used for decades to make high vacuum measurements on a variety of production and laboratory equipment. Interestingly, the manufacturing techniques used to produce these gauges have not appreciably changed during this same time period. Furthermore, the typical, currently marketed extended-range CCIG products involve multiple gauging technologies, which introduce additional complexity and costly design challenges. Our laboratory investigated the impact on gauge cost and performance of a variety of materials,

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manufacturing techniques, electronic circuit designs and sensor technologies that could be employed to produce a reduced cost CCIG that can measure an extended pressure range. The outcomes of this investigation resulted in improved design techniques which have been implemented in a prototype embodiment of a wide pressure range CCIG.

**11:20am VT-MoM10 Operation and Performance of a Wide Range Cold Cathode Ionization Gauge, *Tim Swinney*, *C. Percy*, Pressure & Vacuum Measurement Solutions, MKS Instruments, Inc.**

A novel wide range cold cathode ionization gauge (CCIG), capable of measuring pressures from high vacuum to atmosphere and relying on only one gauge and only one physical electronics mechanism, was recently developed in our laboratory. Many technical challenges exist in operating a CCIG above the standard  $10^{-2}$  Torr upper limit of the current CCIGs available on the market. We will present detailed operational aspects of our new sensor technology including: (1) selection criteria for the discharge characteristics used to derive pressure measurements, (2) accuracy of the pressure measurements produced and (3) long term stability and lifetime of the technology over its wide pressure range.

**11:40am VT-MoM11 Improving Process Resistance of Capacitance Diaphragm Gauges, *B. Andraus*, *C. Strietzel*, *Martin Wüest*, INFICON Ltd., Liechtenstein**

Process industry is constantly changing. New manufacturing processes using new chemistries are developed that can also affect sensors. Yet, quality and cost pressure demand that processes are highly reliable, repeatable and need fewer maintenance interruptions. For capacitance diaphragm gauges, process stability means that process effects on the diaphragm deflection remain in the 1 nm range for a diaphragm with 30 mm diameter. To cope with this we have investigated ways to better protect the CDGs from process related influences. We will present results from experiments performed with protective layers.

## Vacuum Technology

### Room 104C - Session VT-MoA

#### Gas Dynamics, Simulation and Partial Pressure Analysis

**Moderators:** Steve Borichevsky, Applied Materials, Varian Semiconductor Equipment, Ted Martinez, SLAC National Accelerator Laboratory

1:40pm **VT-MoA1 Vacuum System Analysis of a Next Generation Light Source with Synrad and MolFlow+**, *Jason Carter*, Argonne National Laboratory **INVITED**

CERN's SynRad and MolFlow+ vacuum analysis programs continue to be valuable tools for accelerators as vacuum system design challenges increase. The trends for future accelerator vacuum systems, including the APS-Upgrade project, are towards narrower, conductance-limited vacuum chambers which allow for stronger magnets and lower beam impedances but restrict effective pumping and photon shielding. UHV pressure requirements remain fixed or become tighter to increase beam lifetimes and user access which leads to the need for a more thorough vacuum analysis to ensure designs are suitable to many needs. SynRad and MolFlow+ are catered to addressing these challenges and both ease of use and the understanding of their capabilities continues to grow.

SynRad/MolFlow+ users may have been limited in the past by their 3D CAD abilities, however recent improvements to mainstream CAD software such as 'direct modeling' methods have made it easier and faster to build or reverse engineer models with high complexity and precision. This allows for better understanding of complex conductance and quicker iterations on ray tracing schematics. Some examples from the APS will be discussed.

The APS-Upgrade uses SynRad and MolFlow+ extensively for vacuum system calculations and has been digging further into the programs' inputs to build confidence in their predictions. The two programs share a coupling function which predicts dynamic photon stimulated desorption (PSD) outgassing and allows for calculations of dynamic pressures and beam conditioning times. The APS-U is studying this coupling in order to build confidence that their vacuum system design will reach low pressures with reasonable conditioning. Work for this includes studying the sensitivity of the program's inputs and applying the work to existing APS vacuum systems.

2:20pm **VT-MoA3 Simulations of Vacuum Pumping and Beam Conditioning for CHESS-U Vacuum System**, *Yulin Li, X. Liu*, Cornell Laboratory for Accelerator-Based Sciences and Education; *J.S. Mershon*, The College of Wooster

A major upgrade project (dubbed CHESS-U) is planned to elevate performance of Cornell High Energy Synchrotron Source (CHESS) to the state-of-art 3<sup>rd</sup> generation light sources. As a critical part of the CHESS-U project, about 80-m of Cornell Electron Storage Ring (CESR) is to be replaced with double-bend achromat (DBA) lattice to significantly reduce electron beam emittance. In this presentation, we will describe the conceptual design of the CHESS-U vacuum system, with emphasis on the vacuum pumping design and considerations. In the DBA lattice, multifunction dipole magnets with complex magnet poles prevent use of distributed ion pumps as in current CESR vacuum system. Instead, non-evaporable getter (NEG) strips are used to provide distributed vacuum pumping in the dipole vacuum chambers, as well as in the undulator vacuum chambers. Discrete pumps are used in the straights at available spaces between quadrupole and steering magnets. A test-particle Monte-Carlo simulation program, MolFlow+, is employed to evaluate pumping performances of the CHESS-U vacuum system in two aspects. First, we demonstrate that the planned vacuum pumping system can achieve and sustain required ultra-high vacuum level in CHESS-U operations. In addition, we will explore beam commissioning processes of the new vacuum chambers, and simulate the saturations of the NEG strips during the commissioning. These simulations will aid continuing design optimization for the CHESS-U vacuum system.

2:40pm **VT-MoA4 MFIG, A New Vacuum Sensor for Yield Enhancement**, *N.B. Koster, F. de Graaf, Michel van Putten, P.M. Muilwijk, E. Nieuwkoop, O. Kievit, D.J. Maas*, TNO Technical Sciences, Netherlands

This contribution addresses the introduction of a new type of sensor that can disruptively enhance the yield of manufacturing and inspection tools where ultraclean vacuum and control is needed. This includes tools working with high energy photons, ions or electrons.

In 2007, the development of TNO's mass-filtered-ion-gauge (MFIG) started when experts realized that IC manufacturing with EUV needs extremely clean vacuum, while existing sensors are either too slow, expensive or insensitive for real-time monitoring of vacuum cleanliness. The promising results of a "quick-and-clean" test were well-received at the AVS meeting in 2008. Hence TNO incubated the concept in an EU project, in which both instrumentation was improved and application requirements were clarified by interacting with the consortium partners. In 2015 TNO has been awarded a NanoNextNL valorization grant to further mature the instrumentation and prepare for MFIG's market introduction in 2017.

This presentation will update the audience on MFIG's latest performance in laboratory and field tests as well as tell the story how an idea advanced into a product. A short explanation of the technology and new design will be part of the presentation.

3:00pm **VT-MoA5 Dynamic Process Modeling on a Condensation-based Depressurization System**, *Bo Zhang, G. Guo, C. Zhu, Z. Ji*, New Jersey Institute of Technology

A near-vacuum state in an enclosed chamber can be achieved by vapor condensation on a cooling surface. This near-vacuum chamber can function as a vacuum sink for a sustained operation of application that requires depressurization in an open flow environment. To realize such a sustained operation of gas extraction, a complete cycle of regenerating vacuum in the chamber may consist of multiple stages, including a vapor filling process, the vacuum generation by cooling-controlled condensation, a process of gas extraction from depressurization-required application, and a process of flushing non-condensable gas out of the condensation chamber. A dynamic process model is established to describe the thermodynamic characteristics of the entire cycle. The transient and non-equilibrium characteristics in the condensation-induced vacuum generating process is reasonably captured by our computational fluid dynamics (CFD) model, with modified boundary conditions accounting for the complicated coupling mechanisms of heat and mass transfer during the condensation. The CFD simulations for the entire processes are obtained using FLUENT with user-defined functions. In addition, a pseudo-equilibrium-based parametric model is further developed to evaluate various parametric effects for the system design and optimized operation. The CFD simulation results and parametric modeling predictions are partially validated through our experimental measurements.

4:00pm **VT-MoA8 Vacuum Adventures Encountered Towards a Field-Portable Helium Isotope Detector**, *Gary McMurtry*, SOEST, University of Hawaii; *J.R. DeLuze*, Fusion Energy Solutions of Hawaii; *D.R. Hilton*, Scripps Institution of Oceanography, UCSD; *J.E. Blessing*, MKS Instruments **INVITED**

The  $^3\text{He}/^4\text{He}$  ratio in volcanic emissions and dissolved gas in groundwater is often co-seismic with, and sometimes precursory to, volcanic unrest and earthquake activity. Because of the extremely low abundance of primordial  $^3\text{He}$  to radiogenic  $^4\text{He}$ , and difficulties in resolving  $^3\text{He}$  in the presence of hydrogen isobars such as HD, the measurement of this ratio has so far been confined to the laboratory. A field-portable He isotope instrument must overcome these analytical hurdles and be small, compact, lightweight and low enough in power consumption to deploy in critical locations.

We use two compact mass spectrometers, an MKS ion trap and a frequency-modified MKS quadrupole MS, with a full-range pressure gauge and waste pumps based upon noble diode ion or turbo-rough pumping. These are coupled to a high-purity quartz glass port that is heated under high vacuum. Gas samples can be separated from waters or directly analyzed by pumped circulation through a sample chamber. We monitor vacuum quality with the ion trap and use the quadrupole MS to obtain sensitive determination of hydrogen and helium isotopes. Two methods of isobaric separation are utilized: a statistical mass-2 vs. mass-3 regression intercept, and an adjusted (threshold) ionization mass spectrometry (AIMS) technique. Comparison of these two independent methods for 44 data pairs in a "blind collection" after heat ramps to a predetermined maximum temperature are completed yields a significant correlation ( $r = 0.89$ ).

Results on laboratory air are within a factor of 2 of the accepted ratio of 1.40 E-06 ( $R_a$ ). We can obtain the exact air ratio ( $R/R_a = 1.0$ ) if we continuously monitor the MS scans during the heat ramps, allowing for differences in the diffusion rates of  $^3\text{He}$  and  $^4\text{He}$ . With an established power level, keeping to a constant scan time allows air  $^3\text{He}/^4\text{He}$  ratios to be obtained to within 0.1  $R/R_a$ . Adventures in vacuum technology encountered along this developmental pathway include the discovery of temperature-dependent differential diffusion of He isotopes in heated glass, quantum tunneling of  $^3\text{He}$ , amazing enrichments of  $^3\text{He}$  from air, and potential industrial applications of a mass-selective fluid bandpass filter.

# Monday Afternoon, November 7, 2016

4:40pm **VT-MoA10 Use Of A Novel Sensor Using Remote Plasma Emission Spectroscopy For Monitoring And Control Of Vacuum Processes**, *Joseph Brindley, T. Williams, B. Daniel, V. Bellido-Gonzalez*, Gencoa Limited, UK; *F. Papa*, Gencoa USA

Plasma emission monitoring (PEM) has been used for a number of years to either monitor the condition of or actively control vacuum plasma processes. This approach has many advantages such as fast response time, monotonic sensor behaviour and the ability to control uniformity by monitoring different areas of the process. There are however some disadvantages, e.g. there is required a clear line of sight to the plasma that can be obscured by substrate movement, the PEM sensor can become coated by the deposited material and, of course, it can be only be used when the process itself generates a plasma.

A new type of remote plasma generator has been developed, which when combined with advances in miniature spectrometers can be used to perform optical plasma spectroscopy over a wide pressure range of 1 mBar to 1E-6 mBar. Presented are a number of examples of its use as an intelligent pressure gauge (penning pressure measurement in conjunction with plasma spectroscopy), etching process monitoring, vacuum quality monitoring, and reactive deposition control.

A novel, pulsed power, method of enhancing the sputter effect inside the sensor has also been developed. This allows for use of the sputtered cathode emission as a secondary, indirect indicator of the condition of the vacuum and state of the process, enabling monitoring and control of processes otherwise not possible via conventional plasma spectroscopy. Furthermore, this sputter mode of operation has the effect of "cleaning" the sensor's cathode, allowing for extended operation with processes that would otherwise damage the sensor.

5:00pm **VT-MoA11 Calibration of Quadrupole Mass Spectrometers with a Molecular Flow Gas Source**, *Robert Ellefson*, REVac Consulting

In situ calibration of quadrupole mass spectrometers (QMS) after initial calibration is not regularly done. If no calibration gas is available, it requires removal of the QMS from the vacuum system to a calibration test stand where gases and a reference gauge is available. Providing a low-cost calibration gas source dedicated to the QMS enables local calibration checks to qualify the performance of the QMS. This paper describes a molecular flow calibration device that presents known flow rates of gas species to the ion source. The known flow rate produces partial pressures that can be measured with an ion gauge or calculated knowing the conductance of the QMS molecular flow pumping system. The molecular flow in and molecular flow out preserves the gas composition of even a flowing gas mixture prepared in a volume from attached gas sources. The device prepares a known volume of gas ( $300 \text{ cm}^3$ ) at low pressure ( $P_{\text{CDG}} < 10 \text{ Torr}$ ) to assure molecular flow through an orifice with flow proportional to  $C_{\text{N}_2}(28/M)^{1/2}$  for each species. With this small volume, the partial pressure depletes for each species in a predictable manner related to the mass of the gas species. By noting the time elapsed since the valve to the molecular leak is opened, the time-dependent partial flow rate,  $q_i(t)$  of each species is known and a sensitivity  $S_i$  for that species can be calculated as  $I_i(t)/[q_i(t)/C_{\text{out}}]$  from the measured ion current,  $I_i(t)$ . Data showing sensitivity of a QMS as a function of ion source pressures provides information to show stability of sensitivity over a range of pressures. The ability to introduce pure gases and blend or introduce gas mixtures gives conditions to measure QMS accuracy for mixture analysis. The simplicity of the system lends itself to automation of the sensitivity measurement process as the basis for archiving  $S_i$  values over extended periods of time as quality assurance performance data.

## Vacuum Technology

### Room 104C - Session VT-TuM

#### Vacuum Pumping and Material Outgassing

**Moderators:** Martin Wüest, INFICON Ltd., Liechtenstein, Jacob Ricker, NIST

8:00am **VT-TuM1 Applicative Challenges for today's Turbo Molecular Pumps**, *Adrian Wirth, H. Bernhardt*, Pfeiffer Vacuum GmbH, Germany; *N. Cotton*, Pfeiffer Vacuum Inc

**INVITED**

Since its invention in 1958 by Mr. W. Becker, the turbo molecular pump (TMP) has been a milestone in showing new and effective ways of providing oil-free high vacuum. It soon started to replace available pumping principles and as the need for high vacuum grew, it became the standard for modern high vacuum applications. It has since been confronted with increasingly diverse customer needs and performance challenges.

In most aspects, the requirements can be subdivided into primary (pump performance related) properties like pumping speed, gas throughput, compression, fore-vacuum compatibility and secondary ones. The latter including attributes of compactness, ease of system integration, corrosion and condensation insensitivity, usability in areas exposed to ionizing radiation, maintenance friendliness, outgassing and particle cleanliness, emission levels of vibrations, sound or electromagnetic radiation, lifetime and costs of ownership—just to name a few.

From the beginning, demanding applications and processes were on the one hand influencing the vacuum performance of the TMP, hence indirectly affecting the effective shape of the rotor and stator parts and its combination with other pumping principles. On the other hand, triggered by specific and extreme applications or operation settings and surroundings, the secondary requirements gained in importance.

Within this scope, some of today's applicative challenges and customer needs for TMPs will be presented. Examples will include the operation of TMPs exposed to magnetic fields in ion implanters or ionizing radiation at particle accelerators, in magnetic stray field and vibration sensitive systems such as electron microscopes, integrated into systems in analytics where low sound levels and the ease of integration are of special interest as well as in corrosive processes. Multi-port characteristic applications with rotor designs being specifically tailored for the individual vacuum performance require sophisticated calculation know-how. Apart from these operation purposes, the utilization in tool coating, nuclear fusion experiments, XUV lithography and mobile employments impose particular specifications. Furthermore an overview and comparison of process/requirement specific TMP designs will be provided.

The TMP had and still has to meet most diverse applications and hence undergoes a change from purely vacuum performance driven specification inputs into a period where also secondary requirements are ever since gaining in importance and hence impose a continuous evolution of the TMP technology.

8:40am **VT-TuM3 Ion Pump Design for Improved Pumping Speed at Low Pressure**, *Alessandro Abatecola, M. Audi*, Agilent Technologies, Italy

Although ion pumps are widely and mostly used in UHV conditions, virtually every existing ion pump has its maximum pumping speed around 10 E-6 mbar (10E-4 Pa). The discharge intensity in the ion pump Penning cell (the number of ions that bombard the cathode per unit time) is pressure dependent, and it is the main parameter that influences the pumping speed.

A study has been performed to evaluate the influence of magnetic fields and cell dimensions on the ion pump discharge intensity at different pressure. As a result, a combination of parameters has been defined that allows the design and manufacture of an ion pump with maximum pumping speed shifted towards lower pressures.

Experimental results with several different experimental set ups are presented.

A new 200 L/S ion pump specifically designed for UHV operation that incorporates these findings to obtain maximum pumping speed in the 10E-8 mbar (10E-6 Pa), and with all components subject to vacuum firing prior to assembly to obtain the lowest ultimate pressure is described.

9:00am **VT-TuM4 Multi Scaled Titanium Gettered Surfaces for Enhanced Pumping of H<sub>2</sub>**, *Alan Van Drie*, Tri Alpha Energy

The sticking factor for any gas onto a flat surface, whether its physisorbed or chemisorbed can be enhanced by increasing the effective roughness of the flat surface. When done on multiple scale lengths, not unlike a fractal pattern, the sticking factor can approach unity. In the present case, we are interested in pumping H<sub>2</sub> by a fresh coating of titanium, which has a sticking factor of around 3-6% at room temperature on a flat surface. The shape of the fractal pattern and number of scale lengths that can be used is limited by the feasibility of coating titanium on all the exposed surfaces. Using three distinct scale length from ~20 cm, ~2 cm to sub-micron we are able to obtain an estimated 70-80% effective sticking factor. The sub-micron scale is achieved by depositing the titanium onto a cryogenically cooled surface that changes the film's morphology from a dense coating to a porous sub-micron needle like structure. This by itself increases the sticking factor from the normal 3-5% to 20-50%. Some deposition techniques can even create film morphologies with up to 90% sticking factor; eliminating the need for other macroscopic scale lengths. Using these techniques we can create a 2,000 m<sup>3</sup>/s pump for H<sub>2</sub>, with 10 m<sup>2</sup> entrance area that fits within our 15 m<sup>3</sup> vessels. The design principles and preliminary pumping speed and capacity measurements will be presented.

9:40am **VT-TuM6 Outgassing of UHV Stainless Steel Cans**, *Lily Wang, P.D. Honnell*, Los Alamos National Laboratory

UHV stainless steel cans are used to contain material samples in our experiments to study how the materials age under accelerated thermal conditions. In addition to characterizing the material properties after the thermal aging treatment, we also measure the amount of gas released during these thermal aging runs and analyze the off gas collected. In preparing for the experiments, the stainless steel cans were first vacuum-baked at 150°C to remove adsorbed moisture. The vacuum-baked cans were then brought into a dry nitrogen glovebox where the samples were loaded into these cans. The experiments were conducted in static vacuum at various temperatures ranging from 25 to 145°C for a duration ranging from 25 days to several years. The pressure of the gas accumulated in the sample can was measured with a capacitance diaphragm gauge. At each temperature, an empty UHV stainless steel can was also measured. Stainless steel contains dissolved hydrogen that diffuses out and contributes background outgas in vacuum systems. This talk will present the gas evolution results obtained from these empty cans and discuss how this stainless steel outgassing affects our gas evolution measurements.

11:00am **VT-TuM10 Calibration of Reference Samples for Water Vapor Outgassing and Water Vapour Transfer Rate**, *Janez Setina*, Institute of Metals and Technology (IMT), Slovenia; *K. Jousten*, Physikalisch-Technische Bundesanstalt (PTB), Germany

Water vapor is usually main constituent of residual gas in non-baked high vacuum systems. The principal source of water vapor is outgassing from surfaces of vacuum chamber. Significant amount of water vapor is adsorbed on the surfaces every time when the system is vented to moist ambient air. Outgassing rate of water vapor is therefore important parameter of any vacuum material and is needed for proper design of the system to operate in required pressure range.

Traceability of outgassing rate measurements was one of the research activities of the recently finished European project EMRP IND12. For calibration, validation and comparisons of outgassing rate measurement systems different reference outgassing samples were studied. In this presentation we will focus on the reference samples for water vapor outgassing, which were jointly developed and applied for patent by PTB and IMT [1]. For measurement and calibration of outgassing rate from reference samples for different gases, including water vapor, a dedicated vacuum system was developed at IMT. Measurement range of the system for water vapor outgassing rate at room temperature is from 5×10<sup>-7</sup> mbarL/s to 5×10<sup>-3</sup> mbarL/s (equivalent to 2×10<sup>-11</sup> mol/s to 2×10<sup>-7</sup> mol/s, or 3×10<sup>-5</sup> g/day to 0.3g/day). Typical relative uncertainty of measured outgassing rate is below 5 %.

Results of calibration of two water vapor reference samples will be presented. One sample had nominal outgassing rate of water vapor 6×10<sup>-7</sup> mbarL/s (2.5×10<sup>-11</sup> mol/s, or 4×10<sup>-5</sup> g/day) and another sample 3×10<sup>-5</sup> mbarL/s (1.3×10<sup>-9</sup> mol/s, or 2×10<sup>-3</sup> g/day). Both samples represent constant outgassing rate over long period of time. Repeated measurements showed time stability of reference samples better than 3%/year.

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The newly developed water vapor outgassing reference samples can be also used for calibration of measurement instruments for water vapor transfer rate (WVTR).

[1] Patent DE 102014200907 A1 (2015)

**11:20am VT-TuM11 Simulated and Measured Extreme High Vacuum in the Jefferson Lab Polarized Electron Source, Marcy Stutzman, Thomas Jefferson National Accelerator Facility**

The polarized electron source for the Jefferson Lab CEBAF nuclear physics program has stringent vacuum requirements for successful operation. Research projects aimed at improving the static vacuum into the extreme high vacuum range, below  $10^{-12}$  Torr, have investigated outgassing rate reduction through coatings and heat treatments, vacuum characterization optimizing the utilization of commercial extreme high vacuum gauges, and pumping configurations including developments in UHV/XHV cryopumping. Additionally, limitation of dynamic vacuum during operation has been studied using surface analysis and processing toward reducing field emission from the high voltage electrode in the electron source. Both modeling and experimental results of these studies and the impact of incorporating these improvements in the vacuum system for the Jefferson Lab polarized electron source will be presented.

**11:40am VT-TuM12 Outgassing Rate Measurements of 3-D Printed Materials, Makfir Sefa, J.A. Fedchak, J. Scherschligt, National Institute of Standards and Technology (NIST)**

3-D printing of parts has many potential advantages over traditional machining. The outgassing rate of these materials is particularly interesting for determining their performance in a vacuum environment and for other practical applications. We measured outgassing rate of 3-D printed stainless steel and titanium samples. The outgassing rate was measured using pressure rate-of-rise method and throughput method. The outgassing rate of each sample was measured at room temperature. The composition of the desorbed gas was also determined. The experimental results and measurement procedure will be discussed in the presentation.

**12:00pm VT-TuM13 Characterization Studies of UHV Polished Surfaces, Melisa Buie, C. Fields, Coherent Inc; A. Cress, San Jose State University**

Interest continues to remain high in the application of electropolished surfaces. Studies of outgassing and surface treatments dating back to 1969 have remained relevant as science continues to push the lower boundaries of UHV. [1-5] More recently, polished stainless steel surfaces have been tested for use in high voltage applications. [6] The stainless steel flanges must have excellent sealing capability in low compression UHV joints, be able to withstand temperatures above 1200°C, increased corrosion resistance, and provide minimal outgassing.

A characterization study was performed comparing electropolished stainless steel flanges with plated and subsequently hand-polished stainless steel flanges to optimize the flange surface for temperature resistance, corrosion resistance, hermeticity, and outgassing. A randomized designed orthogonal experiment (full factorial) was performed varying the type of polish, the electrolytic exposure time, and post-polish processing temperature. Electroplished surface quality showed a strong interaction between the exposure time to the electrolytic bath and the post-process annealing temperature. Nickel electroplating followed by hand polishing showed resultant plating thickness inconsistencies along with an adverse effect at elevated temperatures.

References:

[1] J. R. Young, J. Vac. Sci. Technol. 6, (1969).

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## Vacuum Technology

### Room 104C - Session VT-TuA

#### Accelerator and Large Vacuum Systems

**Moderators:** Marcy Stutzman, Thomas Jefferson National Accelerator Facility, Marcelo Ferreira, European Spallation Source-ESS, Sweden

#### 2:20pm VT-TuA1 Vacuum Design of the European Spallation Source Target Monolith System, *Peter Ladd*, European Spallation Source, Sweden

INVITED

The European Spallation Source (ESS) is a multi-disciplinary research center based on the world's most powerful neutron source being built in Lund, Sweden. The facility design and construction includes the most powerful linear proton accelerator ever built, helium-cooled tungsten target wheel, state-of-the-art neutron instruments, a suite of laboratories, and a supercomputing data management and software development center. The LINAC will deliver 5 MW of power to the target at 2000 MeV. Ground breaking took place in September 2014 and construction is rapidly progressing towards first neutron on target scheduled for mid 2019.

ESS is a long pulse superconducting linac that accelerates protons in 2.86 ms long pulse stream with a repetition rate of 14 Hz at a 4 % duty cycle providing an average beam power of 5 MW. The stream of protons is intercepted by a helium cooled tungsten target wheel where about 10% are converted to mass, through the nuclear reactions in the spallation process and the remaining 90% is deposited as heat within a distance of about 1m from the target wheel. The moderators and reflectors maximize the yield directing the flow of neutrons to a suite of neutron instruments through neutron guides.

The "Target `Monolith Vessel" (CPMV) is a vacuum vessel nominally 6m in diameter x 9m high fabricated of 304 stainless steel. The major equipment located within this vessel are the helium cooled target wheel, 42 actively cooled neutron beam port inserts that connect to the external neutron guides, actively cooled moderators and reflector plugs and water cooled radiation shielding blocks. The CPMV is designed to operate either under a helium atmosphere, normally at 1 bar pressure, or under high vacuum and is directly connected to the accelerator beam-line that operates under ultra high vacuum conditions. A proton beam window (PBW) physically separates the two environments when the monolith is under a helium atmosphere and in the vacuum mode the PBW is removed and the two vacuum environments are directly connected.

The paper presented reviews the various aspects of the Vacuum Design of the Target Monolith System including material selection, surface finishes and construction issues, equipment sizing and selection and the development of a Strategic Installation and Test Strategy in order to minimize project risk.

#### 3:00pm VT-TuA3 Achievements and Problems in the First Commissioning of SuperKEKB Vacuum System, *Yusuke Suetsugu, K. Shibata, T. Ishibashi, M. Shirai, S. Terui, K. Kanazawa, H. Hisamatsu*, KEK, Japan

The SuperKEKB is an electron-positron collider with asymmetric energies, that is, 7 GeV electrons and 4 GeV positrons, aiming the goal luminosity of  $8 \times 10^{35} \text{ cm}^{-2}\text{s}^{-1}$ . Most of the vacuum components of the main rings (MR), especially in the positron ring, were newly fabricated to manage the high power of synchrotron radiation and the electron cloud effect (ECE), and to reduce the beam impedance, which are essential to keep the low-emittance beams stable in the operation with high beam currents. The construction of the new vacuum system had finished by the end of December 2015, and the beam commissioning started in February 2016. The maximum stored beam currents steadily increased from the beginning, and reached to 650 mA and 590 mA for the positron and electron rings, respectively, by the end of April. The average pressures at these beam currents were on the order of  $10^{-6}$  Pa and  $10^{-7}$  Pa for the positron and the electron ring, respectively. The vacuum scrubbing of the new beam pipes by the synchrotron radiation processed steadily. The pressure rises per unit beam current were on the order of  $10^{-6}$  Pa A<sup>-1</sup> and  $10^{-7}$  Pa A<sup>-1</sup> for the positron and electron ring, respectively. The high gas desorption in the positron ring was due to the electron stimulated gas desorption at aluminum parts in the ring. On the other hand, the reused beam pipes of the electron ring well memorized the surface condition of that in KEKB, which lead to the low gas desorption. The residual gas during the beam operation was continuously monitored using a quadrupole mass analyzer. The main components were hydrogen, methane, carbon monoxide and carbon dioxide. The electron numbers around the positron beam were also

monitored at an arc section in relation to the electron cloud issues. The effect of antechambers and TiN coating on the suppression of electron cloud was confirmed. Newly developed vacuum components, such as the bellows chambers and gate valves with a comb-type RF-shield, and the MO-type flanges with little step inside, worked well as expected. One annoying problem was frequent pressure bursts accompanying beam aborts observed in the positron ring. Discharges at gaps or collision of the beam with dusts were suspected, but the investigation is still in progress. Here the major achievements and problems obtained in the first beam commissioning of the SuperKEKB MR vacuum system are presented.

#### 4:20pm VT-TuA7 Saving Megawatts in a Micron: Tailoring the Surfaces of Superconducting RF Cavities, *Sam Posen*, Fermi National Accelerator Laboratory

INVITED

In particle accelerators, superconducting radiofrequency (SRF) cavities are specially-shaped chambers in which intense electromagnetic fields are built up through resonant excitation, in order to transfer energy to beams of charged particles as they pass through. Large AC currents are generated in the region in which magnetic fields penetrate into the superconductor—just hundreds of nanometers below the surface—dissipating power that must be absorbed in the liquid helium bath that cools the cavities. Because of the significant cost of removing heat at cryogenic temperatures, accelerator scientists take great care in tailoring the surfaces of these superconducting materials to minimize dissipation, as well as to maximize the accelerating electric field. This contribution will present an overview of modern techniques used in SRF surface preparation, including doping with nitrogen, high temperature deposition of tin, and plasma processing. These processes, which generate micron-scale modifications of the surface, will be outlined, and their substantial impact on the accelerator will be presented.

#### 5:00pm VT-TuA9 NEG Coating of Narrow-Gap Insertion Devices and Beam Pipes: Recent Achievements and Future Perspectives, *Paolo Manini, M. Puro, S. Raimondi, T. Porcelli, F. Siviero, E. Maccallini, G. Bongiorno*, SAES Getters S.p.A., Italy

Non-evaporable getter (NEG) coatings are nowadays successfully employed in several accelerator facilities, where stringent ultra-high vacuum (UHV) conditions should be met.

NEG coatings are able to provide high distributed pumping speeds for every getterable gas; in addition, the thermal outgassing and secondary electron yield of a NEG-coated beam pipe are considerably reduced, thus allowing the achievement of better results in terms of base pressure and, consequently, beam lifetime and luminosity.

The use of NEG coatings is especially suitable for narrow-gap beam pipes and insertion devices, which could not otherwise be pumped with the same effectiveness by a series of traditional UHV lump pumps. This aspect is particularly significant in view of next-generation machines, for which long and small-aperture tubes (*i.e.*, down to 4 mm) are envisaged in order to reach even higher luminosities and lower emittances.

Such stringent requirements should be carefully addressed, as a number of technical issues arise—both in terms of coating deposition and characterisation—when dealing with narrow-gap beam pipes of this kind.

SAES' recent achievements in this field are here presented, together with an overview of the ongoing R&D activities, whose aim is to demonstrate the feasibility and pumping effectiveness of narrow pipes under 10 mm of diameter. These include SEM morphological inspections, chemical composition analyses and thickness profiling made by EDX and, finally, measurements of the getter film's sorption capacity for CO. Future perspectives and issues—including the possibility to coat very long and narrow chambers and to perform transmission factor measurements on them—are also reviewed.

#### 5:20pm VT-TuA10 Realisation of a Vacuum System of an EUV Exposure System, *Freerk Molkenboer, N.B. Koster, A.F. Deutz, B.A.H. Nijland, P.J. Kerkhof, P.M. Mulwijk, B.W. Oostdijk, J. Westerhout, C.L. Hollemans, E. te Slighte, W.F.W. Mulckhuysen, M. van Putten, A.M. Hoogstrate, P. van der Walle, J.R.H. Diesveld, A. Abutan*, TNO Technical Sciences, Netherlands

TNO is designing and building an Extreme Ultra-Violet (EUV) exposure facility, as presented last year. This system, called EUV Beam Line 2 (EBL2) will be capable of exposing a wide range of samples, including 6" EUV reticles.

The EBL2 system combines 6 major sub systems;

# Tuesday Afternoon, November 8, 2016

An atmospheric and vacuum handler connected through a load lock, enabling both molecular and particle clean handling of the sample. Samples will be loaded on the atmospheric handler using SEMI standardised EUV dual pods.

The vacuum handler transports the samples to and from all the attached sub systems. The particle cleanliness of the EBL2 system shall ensure that the EUV reticles can re-enter into EUV lithography tools to assess the imaging impact of the exposure after handling and exposure.

The EUV radiation is generated with a Sn fuelled EUV source and focussed with two collectors, providing EUV irradiation on the sample. The two collectors are mounted in a differentially pumped vacuum system which ensures good vacuum quality in the exposure chamber while maintaining the increased pressure in the EUV source.

The exposure chamber is an ultra-clean vacuum chamber which enables exposure of the sample in an ultra-clean environment. The vacuum design also enables a controlled introduction of various contaminants and process gasses to facilitate the customer's request. The sample or EUV reticle is mounted on a clamp that can be moved in XYZ and rotated around X and Z. This movement is achieved with a large hexapod which is located in atmosphere. The vacuum barrier between the hexapod and the exposure chamber is a 1 meter long, CF250 mm edge welded bellow.

The last sub system is an X-ray Photoelectron Spectroscopy (XPS) which is capable of analysing the full surface area of an EUV reticle, as well as performing angle resolved analysis on smaller samples in a specially designed sample holder that can be loaded in the exposure chamber for exposure to EUV.

This presentation will focus on the realisation of the vacuum system of the EBL2 system and will highlight the design choices made to meet the stringent vacuum and particle contamination requirements. Preliminary results of vacuum qualification of chambers will be shown together with progress in building the system.

EBL2 will be publicly accessible as a test facility for EUV lithography related research after qualification, which is expected to be finished end of Q1 2017.

5:40pm **VT-TuA11 Cleaning and Verification Strategies for UCV and UHV Components**, *Michael Flämmich*, VACOM, Vakuum Komponenten & Messtechnik GmbH, Germany; *C. Worsch, S. Gottschall, R. Bauer, U. Bergner*, VACOM Vakuum Komponenten & Messtechnik GmbH, Germany

## INVITED

The requirement of high quality vacuum components for ultra clean vacuum (UCV) and ultra high vacuum (UHV) has become stronger over the last years, especially driven by industrial applications, research institutions and accelerator facilities. Besides the prerequisite of ultra clean surfaces, the outgassing properties from the bulk material are critical for in-situ baked UHV systems. For these applications stainless steel has been and still is the most commonly used raw material. The challenge of suppressing hydrogen outgassing from the bulk material has extensively been discussed in the past. Some approaches seem to be promising, but at the same time they are quite expensive and economically hardly viable. As an alternative to stainless steel, aluminum is regarded as a promising raw material due to some fundamental advantages, even though metal sealed CF components and chambers made from aluminum are hardly available and rarely used.

The present talk focusses on vacuum components and chambers for UCV and UHV applications made from both raw materials, stainless steel and aluminum. In this context, a viable cleaning strategy applying some state-of-the-art cleaning methods will be presented. In order to carefully characterize the extremely low outgassing of components for these vacuum sectors (UCV: non baked; UHV: in-situ baked), appropriate setups for outgassing rate measurements (throughput, accumulation, and pressure rise) will be discussed and respective experimental data will be shown. Measuring, verifying, controlling, and, at the end, knowing the outgassing rate of the produced components enables to explicitly specify, classify and guarantee the cleanliness and outgassing properties of UCV & UHV vacuum components.

As a further focus of the talk, metal-sealed CF vacuum components made from aluminium are introduced. In this context, adequate knife edge stability, complicated weldability and reliable outgassing properties have always been discussed as major challenges. It will be shown that these challenges have been solved lately and that Alu-CF components and chambers (AluVaC®) are today a serious alternative to the established components made from stainless steel.



# Tuesday Evening Poster Sessions, November 8, 2016

## Vacuum Technology

### Room Hall D - Session VT-TuP

#### VT Poster Session (and Student Poster Competition)

##### VT-TuP1 Smart Measurement and Diagnostics Module for Dry Vacuum Pumps, *Wan-Sup Cheung, K. Baik, J.Y. Lim*, KRIS, Republic of Korea

This paper addresses recent industrial demands for more reliable predictive maintenance and diagnostics for the failure protection of dry vacuum pumps operated in the semiconductor and flat display chemical processes. Korean leading companies are very expecting to improve the predictive maintenance and self-diagnostics capability sufficient to meet such higher demands for dry vacuum pumps. This project has started to satisfy Korean industrial demands. On the onset of this work, the first technical issue was to examine what kinds of state variables are measured from dry vacuum pumps. Most of them were found to be the static properties such as body and exhaust temperatures, N<sub>2</sub>-flow rate, motor supply currents of booster and dry pumps, exhaust pressure, etc. Most vacuum pump manufactures have reported that most of vacuum pump failures come from their rotating machinery parts such as rotors, bearings and/or gears. It became apparent that reliable self-diagnostics of vacuum pumps cannot be realized without vibration measurement of those rotating parts. A vacuum pump vibration measurement (VPVM) module has been developed to enable the measurement of three vibration harmonics of rotors, bearings and gears. Tested results of the VPVM module are shown to provide harmonic vibration measurements of rotors, bearings and gears. In addition to the vibration measurement capability of the VPVM module, another technical challenge was to collect the measurements of the state variables available from each vacuum pump via the digital communication interface. This work has attempted to integrate both the traditional state variables and the three harmonic vibration levels into an extended set of state variables required to realize more reliable predictive maintenance and diagnostics of dry vacuum pumps. To obtain the extended state variables, the VPVM module was developed to support three kinds of serial communication ports (RS232C/RS485, CAN and/or SPI) to an individual vacuum pump controller. Furthermore, the VPVM module was designed to provide a 128 MB backup flash ROM to record a time series of the extended state variables in real-time. These records of the extended state variables are used to implement the self-diagnostics and predictive maintenance algorithms of dry vacuum pumps developed and patented by KRIS.

##### VT-TuP2 Vacuum System of Positron Damping Ring for SuperKEKB, *Kyo Shibata, Y. Suetsugu, T. Ishibashi, M. Shirai, S. Terui, K. Kanazawa, H. Hisamatsu*, KEK, Japan

SuperKEKB, which is an upgrade of the KEKB B-factory (KEKB), is a next-generation high-luminosity electron-positron collider. Its design luminosity is  $8.0 \times 10^{35} \text{ cm}^{-2}\text{s}^{-1}$ , which is about 40 times than the KEKB's record. To satisfy tight requirements on beam quality for positron injection, a new damping ring (DR) with a circumference of 135.5 m is constructed in the positron injection system. For beam quality improvement, the positron beam extracted from an injection linac with an energy of 1.1 GeV stays in the DR for 40 ms. Maximum stored current is 70.8 mA, and the number of bunches is 4.

The DR has two arc sections (~110 m) and two straight sections (~20 m). In the arc sections, walls on both sides of the beam pipes are irradiated with synchrotron radiation (SR, critical energy: ~1 keV). To deal with SR and photoelectrons, the beam pipes in the arc sections have antechambers on both sides of a beam channel. The antechamber is also effective in reduction of the beam impedance. The height of the beam channel in the arc sections is 24 mm and the width including the antechambers is 90 mm. To remove the heat by SR irradiation, a water cooling system is also required in the arc sections. In the straight section, on the other hand, an antechamber structure and a water cooling system are not necessary. The cross-section of the beam pipe is octagon with an inscribed circle diameter of 46 mm.

Required beam lifetime due to residual gas scattering is longer than 1000 sec and averaged pressure should be lower than  $1 \times 10^{-5}$  Pa. In the arc sections, pumping speed should be much larger than that in the straight sections because a major dynamic gas load during the beam operation is photon stimulated desorption by SR. Non-evaporable getter (NEG) pumps are mainly used with auxiliary ion pumps, and the average effective pumping speed in the arc sections is about  $0.05 \text{ m}^3\text{s}^{-1}\text{m}^{-1}$  (CO) just after NEG activation. If the photon stimulated desorption coefficient drops to

below  $1 \times 10^{-4}$  molecules photon<sup>-1</sup> by the sufficient SR irradiation (i.e. scrubbing), the target pressure can be achieved even after the average pumping speed reduces by nearly half.

The material of the beam pipes is aluminum alloy. The number of the beam pipes is about 100, and almost all beam pipes are coated with titanium nitride films as a countermeasure against the electron cloud issue. Moreover, the beam pipes in the arc sections have the grooved surfaces on upper and lower sides of the beam channel to reduce the secondary electron yield structurally.

Installation work of the beam pipes will start on May of 2016, and the construction of the vacuum system will finish by the summer of 2017.

##### VT-TuP3 Testing Pump Speed & Thermal Loading of Titanium Arc-Gettered High Speed (~2,000 m<sup>3</sup>/s for H<sub>2</sub>) Cryoboxes, *Ernesto Barraza-Valdez*, Tri Alpha Energy

We have a need for very high pump speed for H<sub>2</sub> and D<sub>2</sub> with a finite capacity that can be periodically refreshed. Special pumps are being developed to achieve S<sub>H<sub>2</sub></sub> ~ 2,000 m<sup>3</sup>/s and S<sub>D<sub>2</sub></sub> ~ 1,500 m<sup>3</sup>/s with an inlet area of 10 m<sup>2</sup> that fits within our 15 m<sup>3</sup> vessel. The pumps are an array of LN<sub>2</sub> (Liquid Nitrogen) cooled cryoboxes with multifaceted surfaces that are coated with titanium using cathodic-arc gettering. Since these are rather large cryoboxes, ~1m<sup>2</sup> inlet area each, the standard AVS test dome pump speed method is not practical in our case. This poster will present our own test methods, which are guided by and checked against simulated pump speeds using Molflow+. Additionally, experimental cooling and thermal stress tests will be presented on the cryoboxes and VCR fittings used to make the LN<sub>2</sub> connections between pumps. This will be compared to our simulated cooling and thermal stress analysis.

##### VT-TuP4 Formation and Characterization of Hydrogenated Amorphous Silicon (a-Si:H) Thin Films Deposited by ECR-CVD with Different RF Powers, *Hugo Alvarez, A.R. Santos, J.G. Fo, F.H. Cioladin, J.A. Diniz*, Universidade Estadual de Campinas, Brazil

Hydrogenated amorphous silicon (a-Si:H) films have been deposited on Si substrates using electron cyclotron resonance (ECR) plasmas. ECR systems are downstream plasma reactors, which allow a separate control of ion energy and ion flux, and can operate at low pressures (1-50mTorr) and can allow reducing sharply ion surface sputtering. A 2.45GHz microwave ECR source generates the plasma at high power (up to 1000W). A 13.56MHz RF power source biases separately the sample chuck. The 2.45GHz ECR source and RF chuck power control the ion flux and ion energy, respectively, allowing low temperature (20°C), low pressure (4mTorr) and low damage chemical vapor deposition (CVD). Due to high discharge power conditions in ECR plasmas, high dissociation degree of silane (SiH<sub>4</sub>), gas molecules can be obtained. SiH<sub>4</sub> diluted in 98% of Ar allows low silane concentration in the ECR plasma that, with high degree of dissociation, can reduce the Si-H bond incorporation in the films deposited. Moreover, to optimize the composition and the microstructure of the deposited a-Si:H films, the H incorporation should be kept at less than 20%, depending on RF power source bias. In this work, to study the effects of RF chuck power in the H incorporation into films, different RF powers of 1, 3 and 5W were deposited on silicon substrates, with fixed parameters: ECR power of 500W, pressure of 4mTorr, substrate temperature of 20°C, gas flows of SiH<sub>4</sub> and Ar, 200 and 20 sccm and 20 minutes. The hydrogen incorporation in the films was determined by FTIR (Fourier Transformed Infrared). These films were annealed at 1000°C, during 60s (Rapid Thermal Annealing (RTA) process). Thus, before and after the RTA annealing, for films deposited with RF powers between 1 and 5W: i) The crystalline level of each film, obtained by Raman spectroscopy, changed from totally amorphous film to amorphous and polycrystalline (19.4% - 32.4%) structures; ii) The images from optical microscopy were used to identify the presence of pin holes on the film surfaces. Using scan profiler system, it was extracted the depth (100µm - 2.2µm) and diameter (1800Å - 90 Å) of them. For that, it can be concluded that if the RF power values increases, the crystalline level increases and pin holes dimensions and densities decreases. Considering that the pin holes are generated due to H incorporation into the films, if RF chuck power is higher, the intensities due to Si-H bonds are reduced (extracted from FTIR analyses), indicating the low H concentration into the films. Thus, the a-Si:H films deposited by ECR-CVD that are part of the hybrid solar cells with a-Si:H-p+/C-Si-n++ structures are going to be presented at the conference.

# Tuesday Evening Poster Sessions, November 8, 2016

**VT-TuP7 Low-carbon Steel Chamber and Double Viton O-ring Sealing for Electron Microscope, In-Yong Park, N.-K. Chung, B. Cho, KRISS, Republic of Korea**

Generally, EM(Electron Microscope) consists of an electron beam generation part, beam control part and a specimen chamber. Among them, in order to get a high imaging resolution, condition and function of electron gun are the most important. Firstly, vacuum level of electron gun chamber is maintained properly depends on the method of electron beam generation. Secondly, gun chamber should be shielded from stray magnetic field which influences the electron beam. Lastly, gun chamber had better have adjustable parts for precise alignment of gun position. In this work, we center around on a simple and cheap electron gun structure for EM. For the purpose of it, we adopt the low-carbon steel for gun chamber material and double Viton O-ring for gun chamber sealing, thereby succeeding in making adjustable gun system maintaining UHV(Ultra High Vacuum) condition.

Recently, B. Cho shows that Low-carbon steel ( $C \leq 0.2$  wt. %) has a sufficiently low outgassing rate for constructing UHV chamber [1]. Low-carbon steel is a soft magnetic material that is relatively inexpensive and has a high magnetic permeability, so it block out the stray magnetic field. O-ring is usually used for motion vacuum part and also possible to align electron gun in high vacuum. Schottky emitter and cold field emitter are used for high resolution EM, however those require the UHV condition for preventing contamination of tip surface. For EM based on Schottky emitter, we designed the electron gun with low-carbon steel and double O-rings which support UHV condition by pumping out the permeated gases between double O-rings. We compared the performance of double Viton O-rings and double Kalrez O-rings experimentally after 150 °C baking process. The Viton material is better than Kalrez in our system and the vacuum pressure of electron gun arrived at  $\sim 10^{-8}$  Pa at room temperature. The vacuum pressure is maintained as UHV when the electron gun chamber surface is moved at the double O-ring surface. We applied the Viton double O-ring electron gun system to commercial EM replacing original electron gun to get EM images. We observed the evaporated gold nano-particles and got the magnification up to 200K. Also, we measured the electron beam current stabilities of extractor and probe beam. We demonstrated that Low-carbon steel and double Viton O-ring can be used for EM with Schottky emitter and show highly magnified images of gold nano-particles. We anticipated that these methods could replace the existing electron gun system of EM, thereby providing a simple structure and reducing the cost of production of EM.

[1] C. Park, T. Ha and B. Cho, J. Vac. Sci. Technol. A 34(2), (2016).

# Wednesday Morning, November 9, 2016

## Vacuum Technology

### Room 101D - Session VT-WeM

#### Vacuum Technology – History and Innovation (8:20-10:00 am)/Transfer and Manipulation (11:00 am-12:20 pm)

**Moderator:** Jay Hendricks, National Institute of Standards and Technology

##### 8:20am VT-WeM2 A New Approach to Vacuum Technology Education at a Distance, *Del Smith, N. Louwagie*, Normandale Community College

Education and training for technicians who work in the vacuum and vacuum coating industries is becoming more critical as the complexity of the equipment and processes increases. In addition, many of those currently working as technicians are approaching retirement age, with no obvious source for the large number of replacement personnel that will be needed. Many technicians will benefit from a formal educational program giving them grounding in the basic sciences and developing their soft skill set. Several local companies utilizing vacuum and coating technologies partnered with Normandale Community College in 1998 to develop a formal educational program to train technicians in the basic concepts of vacuum and thin film technology. Recently, the need for the expansion of this program to geographic areas that are currently underserved and cannot support a college program in these specialties became apparent. Normandale Community College in Bloomington, MN, is in its second year of a three year project, funded by the National Science Foundation, Division of Undergraduate Education, Advanced Technological Education. The focus of this project is to develop the vacuum technology curriculum, processes and equipment needed to train technicians at locations remote from the primary instructor. This paper presents the current status of this project, with details about curriculum development, remote classroom technology and the development and use of advanced vacuum training hardware systems for use at the remote learning site that include features not previously available.

##### 8:40am VT-WeM3 It's All Because of the Vacuum..., *H. Frederick Dylla*, American Institute of Physics **INVITED**

A number of key scientific demonstrations from the 18<sup>th</sup> century to the present were enabled by the essential task of obtaining a low enough level of vacuum. This talk explores a number of well-known events in the history of science and technology that depended on achieving a remarkable level of high vacuum for the era contemporary to the demonstration. We start with Franklin's lyceum experiments where he applied static voltages across glass cylinders where pressures were lowered below ambient conditions with crude air pumps. This work presaged subsequent work on gas discharges and modern accelerator cavities. A century later, J. J. Thomson was the first to make an electron beam by lowering vacuum levels sufficiently to prevent ionized residual gas ions from shielding the negative particle beam. Fast-forward to the early 1960's where G.K. O'Neill invented the now standard configuration for contemporary particle physics experiments- the high intensity storage ring. This configuration demanded and achieved true ultrahigh vacuum levels on an industrial scale. The story concludes with this year's remarkable detection of gravity waves using LIGO - the kilometer-scale laser interferometers that required extreme high vacuum levels for all residual gas components.

##### 9:20am VT-WeM5 The Next Generation Quantum-based Metrology for Miniaturized Sensors and Standards, *Gregory F. Strouse*, National Institute of Standards and Technology **INVITED**

Development of innovative sensors and standards that—through improvements in cost, size, speed, and durability—will enable new manufacturing processes, tools and products of tomorrow. The NIST vision is that these innovations will improve the dissemination of standards to the point where routine exchange of artifacts for measurement quality assurance is no longer needed. Quantum and photonic based rugged small-scale vacuum sensors open new horizons in measurement science and represent a paradigm shift in how metrology is done. Networks of small and precise sensors embedded within structures and composite materials could improve their performance and reliability. These sensors draw upon a range of technologies not previously exploited for these applications, such as nanofabrication, photonics, and atomic physics. Photonic and quantum-based vacuum sensors will allow both the absolute sensing of vacuum and the realization of the SI at the user site and will allow the user to calibrate other sensors or directly measure process vacuum levels for critical applications. Several related research programs at NIST are geared towards realizing the vision of small or chip sized absolute sensors for

practical applications. NIST is building a sensor program, with the goal to establish a set of chip-scale tools that enable real-world use. An example is the chip-scale cold-atom technology requires maintaining UHV conditions throughout the operating lifetime of the device, posing practical technical challenges to the vacuum engineering. Our program to replace primary mercury manometers with photonic-based primary standard relies on lasers and Fabry-Pérot optical cavities that are stable from several atmospheres of pressure down to high-vacuum. These cavities will be field-deployable absolute sensors requiring no calibration. Other quantum and photonic based sensors include dynamic pressure measurements and thermometry. These programs will be discussed in terms of the larger programmatic view of how quantum-based, chip scale technologies will disrupt vacuum technology, primary standards, and pressure, vacuum and temperature measurement.

##### 11:00am VT-WeM10 Vacuum Transport for Realization and Dissemination of the Redefined Kilogram at NIST, *Eric Benck, E. Mulhern*, NIST **INVITED**

In 2018 the unit of the kilogram will be redefined. Instead of being based on the physical artifact known as the International Prototype Kilogram (IPK), the kilogram will be defined by fixing the value of a fundamental physical constant, Planck's constant. It will now be realized using watt balance or x-ray crystal density (Avogadro) experiments. This will enable any research group throughout the world to realize the unit of mass if they have sufficient technical expertise and equipment. Both of these experimental methods are optimally designed to operate in vacuum. As a result, mass metrology must now deal with the issues of maintaining, manipulating and moving masses under vacuum.

At the National Institute of Standards (NIST), at least five different experimental systems operating in vacuum will be used for the realization and dissemination of the kilogram. First, the kilogram will be realized using the NIST-4 watt balance. Second, NIST is developing a unique apparatus called the magnetic suspension mass comparator (MSMC) to transfer the unit of mass in vacuum to a mass in air. Third, a commercial vacuum comparator will be used to directly compare different masses in vacuum and for sorption studies. Fourth, a new plasma cleaning station is being developed to use a downstream hydrogen plasma source to clean a mass. And finally, there will be a vacuum mass storage facility where multiple calibrated masses can be kept for later mass comparisons.

In order to transport a mass under vacuum between the different apparatus, a custom built mass transport vehicle (MTV) has been constructed. It is essentially a mobile vacuum chamber made out of a 4 way cross and gate valve. It has a wide range pressure gauge and a battery powered getter pump. The vacuum chamber is supported by an aluminum frame mounted on castors. The MTV can maintain a vacuum below  $1.3 \times 10^{-3}$  Pa for 30 minutes without pumping which is sufficient time to transport a mass between the different rooms housing the experimental apparatuses. The MTV can be attached to a load lock on each apparatus which can extract the mass and transfer it inside while maintaining the mass in vacuum. The complete transfer process of a mass from one machine to another with the MTV takes on the order of an hour. Most of this time is due to the evacuation time of the load lock after the MTV has been attached.

##### 11:40am VT-WeM12 Handling, Transfer, Storage, and Shipping of Commercial Thin Film Hydride Disk Target Samples, *James Provo*, J. L. Provo, Consulting

Handling, Transfer, Storage, and Shipping of Commercial Thin Film Hydride Disk Target Samples

James L. Provo\*

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

Thin film hydride targets are important for many applications including, accelerator research, various neutron devices, contraband detection, etc.

They are very sensitive to air-oxidation and easily contaminated by improper

handling. Air-exposure, which oxidizes Group IIIB, IVB, and rare earth film materials, affects their operating properties. This paper will discuss the

development of handling techniques, and special transfer and shipping containers for hydride target samples from post processing to transfer and shipment to a customer. Studies were performed to determine the best physical

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handling devices, procedures for reducing particulate contamination, and for reducing air-exposure and moisture from samples before actual use. Initially, as an example, samples in an air-exposure hydriding system, were backfilled with an inert gas just before opening into an environmentally controlled clean room, quickly moved to an inert gas glove box, and then placed in special vacuum transfer or shipping containers, as quickly as possible, and then pumped down to a vacuum of  $\sim 1 \times 10^{-7}$  Torr(  $1.33 \times 10^{-5}$  Pa) or less. For optimum handling conditions, a system was developed with the major components being a hydride loading system in a double-sided stainless steel glove box contained in an environmentally controlled room. This glove box contained a loader vacuum chamber, a video microscope, a HEPA filter/ fan module and a hydriding gas manifold. The glove box had an inert Ar or N<sub>2</sub> atmosphere, achieved by circulating the gas through a commercially made purifier which striped oxygen and water vapor. The glove box atmosphere was monitored by an oxygen monitor, and a water vapor analyzer. When loaded samples are removed from the chamber of such a system, samples are automatically in a pristine environment,with very low particulate contamination, and a minimum amount of water vapor. On the other side of the glove box, samples are placed into transfer and/or shipping containers, which are then pumped down to high vacuum conditions for shipment. Examples of sample handling clips, and sample containers are given. Results have shown, that by using such methods and techniques, hydride target disk samples can be successfully processed, handled, transferred and shipped in a condition very close to that as processed out of a loader.

\* Formerly, Principle Member of the Technical Staff at Sandia National Laboratories,  
Albuquerque, New Mexico 87185 (Retired); electronic mail:  
jlprovo@verizon.net

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