Vacuum Technology Division
Room A213 - Session VT-MoM

Pumping, Outgassing, leaks, and Vacuum Pressure Measurement

Moderators: Scott Heinbuch, MKS Granville-Phillips Division, Longmont, Giulia Lanza, SLAC National Accelerator Laboratory

8:20am **VT-MoM1 Operational Experiences of Compact Non-Evaporable Getter Pumps in CHESS-U and CBETA***, **Yulin Li**, Y. Lushtak, L. Ying,* Cornell University

In two recently commissioned accelerator projects at Cornell Laboratory of Accelerator-based Sciences and Education (CLASSE), a large number of high pumping capacity, compact non-evaporable getter (NEG) pumps were implemented to fulfill the required vacuum performances with very tight space constraint. In the Cornell Brookhaven ERL Test Accelerator (CBETA), NEGs are the only installed pumps. At a maximum electron beam energy of 150-MeV, no beam-induced gas load is expected in normal beam operations. We have demonstrated that adequate level of vacuum ($P<10^{-8}$ torr) can be achieved quickly after a ultra-dry nitrogen venting without in situ bakeout, which provide required flexibility in the CBETA vacuum system for various beam test configurations. In contrast, very high dynamic gas-load due to synchrotron radiation induced desorption (SRID) is expected in the vacuum system for the CHESS-U Upgrade, a major upgrade project for the Cornell High Energy Synchrotron Source (CHESS). During the commissioning phase of the CHESS-U, an extremely high SRID gas load may not only cause rapid NEG saturation (thus requiring frequent NEG re-activations), but also may potentially damage the small sputtering ion pumps (SIPs) of the NexTorr® (a NEG-SIP combination pump from SAES Getters). Protective control program is developed to prevent the potential damage to these SIPs, while keeping monitoring. In this paper, we will present our operational experiences of these compact NEGs in both CBETA and CHESS-U projects.

8:40am **VT-MoM2 Al_2O_3 Coated Stainless Steel Vacuum Chamber and Parts***, **Martin Wüest**, Y. Kuzminykh, G. Mata Osoro, W. Fuchs, J. Gabathuler, L. Ospelt,* INFICON Ltd., Liechtenstein

We built a vacuum system for calibration of total pressure sensors in the conventional way. We made initial performance measurements such as pumpdown times and achieved base pressure. This system was then taken apart and the stainless steel parts (chamber and fittings) were then coated with an Al_2O_3 layer using an ALD process. To do this we cleaned the parts with ozone, heated them to 300 °C and then finally coated them with an Al_2O_3 ALD process. We rebuilt the vacuum system using those coated parts and performed the performance test again. The result is that we can achieve a lower base pressure in shorter time. The achieved base pressure is approximately a factor 3 lower in the coated version compared to the uncoated version.

9:00am **VT-MoM3 Comparative Outgassing Study of Identical Vacuum Chambers***, **James Fedchak**,* National Institute of Sandards and Technology (NIST)

We have measured and compared the H_2 and water outgassing rates for 7 identical vacuum chambers constructed of common vacuum materials and heat treatments: 304L, 316L, 316LN-ESR (electro-slag remelt), titanium, aluminum vacuum-fired 316L, and vacuum-fired 316LN-ESR. These chambers are of identical geometry and are from the same manufacturer. Comparison studies of outgassing from a large selection of chambers has the advantage over those of single samples or chambers in that the influence of chamber geometry in minimized. Much of the motivation for NIST to conduct this study is to identify ultralow outgassing materials for UHV and XHV vacuum systems, common candidate materials include aluminum, titanium, and vacuum-fired 316L or 316LN-ESR stainless steel. Obtaining these low pressures usually requires vacuum chambers with outgassing rates much less than 10^{-9} Pa L s^{-1} cm^{-2}. In addition, vacuum chambers constructed from materials with ultra-low outgassing rates can help reduce the cost of large vacuum systems by requiring fewer pumps (with the associated cost of operation and maintenance) to obtain the desired ultimate pressure. Other considerations in the selection of vacuum materials include the material cost, strength, machinability, weldability, and chemical resistance. One of our aims of this study is to put outgassing rates into the engineering tool kit. In addition to the above materials, we intend to present data on post process of some of the chambers, including electropolishing and a light air-bake, and on mild steel chambers.

9:20am **VT-MoM4 The NIST VAcuum LEaks System (VALES): a new system for the primary and comparison calibration of small gas flows.***, **Julia Scherschligt**, J.A. Fedchak, R. Vest,* National Institute of Sandards and Technology (NIST)

Helium leak standards for low molecular flow rates are critical to the calibration of leak detectors, gas analyzers, and other equipment used in vacuum, aerospace, and space industries. Since 1984, the National Institute of Standards and Technology (NIST) has provided calibration services for the calibration of vacuum leak standards using two independent systems: The Primary Leak System (PLS), which calibrates leak artifacts against a flow meter, and the Leak Comparison Standard (LCS) which compares a customer leak to a NIST owned leak artifact as a function of temperature. The PLS system has recently been upgraded with a new mass-sensitive detector (a quadupole mass analyzer or QMA). The LCS has been retired from service but its functionality has been efficiently replicated on PLS. In this talk, we will describe the new calibration system, VALES, present recent results on the characterization of the new PLS detector and discus the on-going upgrade and automation of the entire calibration system

9:40am **VT-MoM5 Creating a Controlled Gas Environment for Lifetime Testing of EUV Optics***, **Timo Huijser**, M. van Putten, M.J. van der Lans,* TNO, Netherlands

Optics used in Extreme ultra-violet (EUV) lithography typically operate in an environment of 0.01 to 0.1 mbar hydrogen. Since EUV machines cannot receive a bake out after installation it is difficult to reduce background outgassing such as for water, oxygen, nitrogen and hydrocarbons. Although the partial pressures of these contaminants are orders of magnitude lower, their presence in combination with EUV irradiation induces oxidation, etching, deposition and other processes that affect the lifetime of EUV optics.

At TNO these processes are studied using an EUV beam line facility (EBL2). To enable this type of research a method was developed to create a well-controlled environment of multiple gases with defined and stable partial pressures.

The gas environment typically consists of hydrogen (10^{-2} to 10^{-1} mbar) with added oxygen, water, nitrogen and/or hydrocarbons (10^{-8} to 10^{-4} mbar). The strategy for controlling the gas environment is to start by setting the pressures of the additives prior to adding hydrogen. For accuracy, the pressure values are chosen such that the pump speed drop after adding hydrogen is taken into account. In order to do this, the pump speed of all gases needs to be known for both (ultra) high vacuum conditions as well as medium vacuum conditions (after addition of hydrogen). The procedure for setting the gas environment comprises 4 steps:

1. Determine the pump speed at HV conditions of relevant additives such as oxygen, water, nitrogen and/or hydrocarbons
2. Calibrate the differentially pumped RGA system with a gas mixture of hydrogen with defined, small fractions of additives.
3. Inject this mixture in the exposure chamber at nominal operating conditions. Using the RGA calibration data, the individual pump speed of each species in hydrogen is now calculated.
4. Set the partial pressures of additives prior to adding hydrogen, taking into account the calculated drop of pump speed.

Once step 1 to 3 have been carried out for all additives the resulting pump speed values can be applied for each exposure as long as the vacuum system and its geometry are not altered. As a result the partial pressures can be set quickly using common ion gauges.

The method developed to create a well-controlled environment of multiple gases along with its corresponding procedures and results will be presented.

10:00am **VT-MoM6 Sampling System Design to Predict Mixture Composition at a Quadrupole Mass Spectrometer Ion Source***, **Robert Ellefson**,* REVac Consulting

The use of gas mixtures to measure the sensitivity and fragmentation ratios for calibration of a quadrupole mass spectrometer (QMS) simplifies the process and enables *insitu* calibrations. A necessary factor in a mixture calibration method is knowledge of the composition present in the QMS ion source and at the reference ion gauge. Under the conditions of molecular flow of the mixture into the ion source and molecular pumping of the outlet flow, the composition at the ion source is equal to the stated values of the mixture. This knowledge enables a composition-weighted

correction of the ion gauge reading to get the true ion source pressure due to different gauge sensitivities of each species. The partial pressures of components in the ion source are calculated using the mole fraction of the mixture's species times the corrected ion gauge pressure. The sensitivity for a specific gas species is the ratio of the ion current representing the species to the calculated partial pressure at a common time.

The gas dynamics of very low flow rate gas into the ion source from an end crimped capillary or small pore size frit from a high-pressure mixture is presented. The low flow rate produces molecular effusion and the stated mixture composition is established in the ion source. Another model for low pressure (< 10 Torr) gas introduction through an orifice predicts molecular flow into the ion source and a (correctable) species dependence of the mixture composition as a function of time as the species deplete from the sample volume. Results of QMS calibrations using these gas sources and methods are presented together with composition analyses of unknown gas samples.

10:40am **VT-MoM8 Quantum Pressure Standard in the range 200 Pa to 20 kPa using Superconducting Microwave Cavity**, ***Laurent Pitre,*** LNE Cnam-LCM, France*; P. Gambette,* LNE-Cnam LCM, France*; R.M. Gavioso, D.M. Ripa,* INRiM, Italy*; M.D. Plimmer,* LNE-Cnam LCM, France **INVITED**

An LNE-INRiM collaboration is conducting proof-of-principle tests of a primary pressure standard operating at pressures 200 Pa< p < 20 kPa at temperatures 4.6 K < T < 5.8 K. The proposed standard is based on precise measurements of the microwave resonance frequencies of a quasi-spherical, helium-filled, superconducting cavity maintained at cryogenic temperatures. Ultimately, the accuracy of this standard will be competitive with the present standard at LNE over the whole pressure range. The proposed standard exploits 4 theoretical and technological advances: (1) recent *ab-initio* calculations of the microwave-frequency refractive index of helium $n(p, T)$ near 6 K with an uncertainty corresponding to a relative pressure uncertainty $u_r(p) < 1.10^{-5}$ (at a 68% confidence level); (2) the commercial availability of cryogen-free, low-cost, pulsed-tube refrigerators, (3) the ability to manufacture superconducting microwave cavities with resonance quality factors on the order of 5 million (4) and the impending change of the SI that fixes the value of the Boltzmann constant, thereby reducing the uncertainty of thermodynamic temperature determinations in the cryogenic range. A crucial requirement for microwave pressure standards is maintaining the purity of the ^{4}He sample under test. For the proposed standard, this is facilitated by cryogenic cold traps that effectively remove all impurities except ^{3}He.

The first experimental result will be present with a 2.5 cm radius and with Nobium coated quasi sphere. A particular focus on the hydrostatic head correction and the thermomolecular effect will be presented during the presentation.

11:20am **VT-MoM10 Progress Toward Primary Pressure Measurements based on Refractive Index**, ***Kevin Douglass****, J.E. Ricker, J. Hendricks,* National Institute of Standards and Technology (NIST)

Towards the goal of quantum-based traceability of the Pascal, NIST has developed an optical pressure measurement system where traceability is achieved through accurate quantum mechanical calculations of the refractivity virial coefficients. Extremely accurate measurements of refractive index are possible; however, traceability is currently through the mercury manometer. Primary traceability for the NIST Fixed Length Optical Cavity (FLOC) will require an independent approach, relying solely on refractive index type measurements. A critical step in achieving primary traceability for the FLOC is determining the pressure dependent distortion terms. This is a major challenge because the pressure and the distortion term need to be determined simultaneously. A dual-wavelength approach can provide two measurements in-order to solve for the two unknown variables. The operational wavelengths for this measurement are at 633 nm and 1542 nm. The dual-wavelength approach and current results will be presented.

11:40am **VT-MoM11 Application of Porous Conductance Element for High Vacuum Gauge Calibration**, ***Martin-Viktor Johansson****,* Aix Marseille University, France*; M.P. Wüest,* INFICON Ltd., Liechtenstein*; P. Perrier,* Aix Marseille University, France*; I. Graur,* Aix-Marseille University, France

It is well known that the sensitivity of high vacuum gauges, such as ionization gauges (IG), drift in time [1] and need to be periodically recalibrated. Removal of the sensors from in-situ is time-consuming, and calibration by direct comparison with calibrated IGs has limited accuracy.

The permeability and conductance of the micro sintered stainless-steel membranes with pores varying from 0.2 µm to 0.5 µm was investigated for a wide range of pressure and several gases, from continuum to free molecular regime using a previously developed method [2]. The conductance of this kind of membranes was found constant for low pressures. This property makes the studied membranes particularly suitable as a leak element, by taking advantage of the constancy of conductance in the free molecular regime.

Effective pumping speed of a turbo molecular pump and the conductance of the fabricated microporous conductance element tend to a constant value as the pressure decreases [3], which is still in the measurement range of a 10 mTorr CDG. With the measured constant value, we can use a CDG and the sintered porous stainless steel to calibrate high vacuum sensors. The proposed configuration can be put on the user's high vacuum system for calibration on site. This calibration method is based on absolute pressure sensors and was found to be robust and easy to use.

References:

[1] H.Yoshida, K.Arai, H. Akimichi, M. Hirata, Stability tests of ionization gauges using two-stage flow-dividing system, Vacuum, 84, 705-708, 2009

[2] M.V. Johansson, F. Testa, I. Zaier, P. Perrier, J.P. Bonnet, P. Moulin, I. Graur, Mass flow rate and permeability measurements in microporous media, Vacuum, 158, 75-85, 2018

[3] H. Yoshida, K. Arai, M. Hirata, H.Akimichi, New leak element using sintered stainless steel filter for in-situ calibration of ionization gauges and quadrupole mass spectrometers, Vacuum, 86, 838-842, 2012

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