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

Progress with Measurement in Vacuum
Moderators: Martin Wüest, INFICON Ltd., Liechtenstein, Steve Borichevsky, Applied Materials, Varian Semiconductor Equipment

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

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

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

Accepted: May 2016, Issue 5, 10.1063/1.4949504

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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