Monday Morning, November 10, 2014

Vacuum Technology
Room: 303 - Session VT-MoM

Vacuum Measurement, Calibration, and Primary Standards
Moderator: Steve Borichevsky, Applied Materials,Varian Semiconductor Equipment, Yulin Li, Cornell University

8:20am **VT-MoM1 Miniature Fiber Optic Pressure Sensors: Technologies and Applications**, ***Miao Yu***, University of Maryland, College Park **INVITED**

Compared with their electrical counterparts, fiber optical sensors offer many advantages including small size, immunity to electromagnetic interference, convenient light guiding through optical fibers, high sensitivity, high resolution, large bandwidth, and low noise. Recently, miniature fiber optic pressure sensors have attracted much attention for many applications including biomedical, surveillance, and industrial applications. Most of these sensors are based on a Fabry-Perot interferometer fabricated directly on an optical fiber end face with a silica/silicon (Si) diaphragm serving as a pressure transducer. Although these sensors have a small size (~100s micros in diameter), they suffer from important issues including low sensitivity due to the high elastic moduli of silica/Si (130-185 GPa), and high brittleness and easy breakage of the sensor elements.

In this paper, the research program at the Sensors and Actuators Laboratory (SAL) of the University of Maryland on miniature fiber optic pressure sensors based on polymer or grapheme diaphragms will be introduced. First, our work on polymer based miniature optical pressure sensors will be discussed. The polymer materials have superior elasticity and high fracture strength, which enables polymer based sensors to have superior sensitivity even at a small size and helps prevent cracking or breaking of the sensors. Furthermore, these sensors can be fabricated by using relatively simple and inexpensive processes. We have developed several unique low-cost micro-fabrication processes for these sensors, including self-aligned photolithography and UV molding process. By using simple and safe procedures, a polymer based Fabry-Perot cavity can be directly fabricated at the end of optical fiber, thus eliminating the necessity for complicated assembly of the sensing element and the optical fiber. Further, since polymer based sensors inherently suffer from temperature driftdue to the large thermal expansion coefficient of polymer materials, novel temperature compensation methods for the polymer based fiber optic pressure sensors will also be discussed. Second, miniature fiber optic pressure sensors utilizing a graphene diaphragm will be presented. Graphene is believed to be one of the strongest materials and the thinnest film in the universe, and it can be stretched by as much as 20%. These unique mechanical properties render graphene an excellent choice for miniature acoustic sensors with unprecedentedly high sensitivity, large bandwidth, and large dynamic range. Finally, the potential applications of these different sensors will be discussed.

9:00am **VT-MoM3 Quantum Based Vacuum Standard**, ***Jay Hendricks***, *J.A. Stone, J.E. Ricker, P.F. Egan, G.E. Scace, D.A. Olson,* National Institute of Standards and Technology, *D.R. Gerty,* Sandia National Laboratories, *G.F. Strouse*, National Institute of Standards and Technology

The future of pressure and vacuum measurement will employ lasers, Fabry-Perot optical cavities, and quantum physics. Photons interact at the quantum level with matter such that light travels at a slower speed in gas than it does in vacuum. NIST is developing a fixed length optical cavity (FLOC) and variable length optical cavity (VLOC) that will make simultaneous ultra-precise measurements of vacuum and gas cavity photon-path-lengths. While pressure is a widely measured unit in every day processes, the standard on which it is based, the mercury manometer is quite old and traces its early beginnings to 1643. In the future, the mercury barometer will be replaced with a new standard based on quantum chemistry calculations of helium's refractive index. This will enable the replacement of all artifact-based mercury standards. Measuring pressure optically represents a paradigm shift in the way the unit is realized and will move us from a primary standard based on an artifact to a primary standard based on quantum-chemistry calculations of helium's refractive index. This talk will cover current status and early prototype results of NIST's Innovations Measurement Science (IMS) project (the second year of five) that will have profound impacts on how pressure, temperature and length in air measurements are made in the future. While the primary aim of the project is to create new measurement infrastructure for NIST, it will also create exciting spin-off technology that will have large impacts for US manufacturing and world metrology.

9:20am **VT-MoM4 New PTB Standard to Provide Traceability for Partial Pressure Measurement**, ***Karl Jousten***, Physikalisch-Technische Bundesanstalt (PTB), Germany

Partial pressure measurement in vacuum by quadrupole mass spectrometer (QMS) is an important tool to control and monitor processes in industry and to measure outgassing rates. It is, however, difficult to obtain reliable results with quadrupole mass spectrometers, since its calibration is ill-defined and at present there is no traceability to any national primary standard.

PTB has recently established a new calibration system to calibrate QMS. It is based on the continuous expansion method and allows generating three partial pressures at the same time in the calibration chamber with pressure ratios between them of a factor of up to 10^8 with the uncertainty of each partial pressure depending on the value and the gas species. In the minimum the uncertainty of partial pressure is close to 1%. With less accuracy more gas species than three can be mixed.

The flow of desired gas into the calibration chamber is generated by nano-holes, glass capillaries or sintered elements characterized in terms of conductances for some gas species. Since the flow through these conductance elements is of molecular type up to about 10 kPa, the flow can be predicted for any gas species.

Four QMS were characterized with the new system and some results will be given. The first goal with this standard is to test and establish calibration procedures for written standards for several parameters of QMS like sensitivity, minimum partial pressure and minimum concentration.

Support through the EMRP IND12 project is gratefully acknowledged. The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.

9:40am **VT-MoM5 Study of Long Term Stability of Quadrupole Mass Spectrometers**, ***Janez Setina***, Institute of Metals and Technology (IMT), Slovenia, *A. Elkatmis, R. Kangi,* Ulusal Metroloji Enstitüsü (TUBITAK UME), Turkey, *K. Jousten*, Physikalisch-Technische Bundesanstalt (PTB), Germany, *M. Bergoglio,* Istituto Nazionale di Ricerca Metrologica (INRIM), Italy, *F. Boineau*, Laboratoire National de métrologie et d'Essais (LNE), France, *S. Ruiz,* Centro Español de Metrología (CEM), Spain, *M. Vicar*, Czech Metrology Institute (CMI), Czech Republic

Quadrupole mass spectrometers (QMS) are widely used in industry for leak detection, residual gas analysis and measurements and control of gas composition in vacuum processes, which require well defined partial pressures of different gases in low pressure environment. Consistent measurements and process stability are possible solely if measurement instrumentation is sufficiently stable with time. Only few studies about the time stability of QMS have been reported in the literature. They mainly indicate that QMS instruments are less stable than typical Bayard-Alpert ionization gauges, so frequent recalibrations may be required.

To get information about typical quality of commercial instruments, a group of European national metrology institutes performed a joint study of time stability of some metrological characteristics of seven different QMS. Parameters under study included: sensitivity for gases He and N_2, mass resolution, mass scale stability, secondary electron multiplier (SEM) gain, and minimum detectable partial pressure. Typical check intervals were 3 months. Study started in the middle of 2012 and the overall duration was two years.

In 3 months periods the typical changes of sensitivity between 10% and 30% were observed. The peak positions were stable within 0.05 amu and 0.2 amu and mass resolution was stable within 0.02 and 0.05 amu. For most of instruments a gradual decrease of the SEM gain as a result of aging of the multiplier was observed. For some instruments the SEM gain dropped by more than 40 % in 2 years period.

Support through the EMRP IND12 project is gratefully acknowledged. The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.

10:00am **VT-MoM6 The Stability of Spinning Rotor Gauges as Transfer Standards**, ***James Fedchak***, National Institute of Standards and Technology (NIST)

The spinning rotor gauge (SRG) has long been used as a transfer standard for high vacuum calibrations and in national and international intercomparisons of high-vacuum standards. It is typically used over a pressure range of 1×10^{-4} Pa to 1.0 Pa (10^{-6} Torr to 10 mTorr) and tends to be favored by metrology and calibration laboratories because of its reputation as being a very stable standard. Here we will review the stability data for SRGs used in international key comparisons. Different rotor materials and various transfer techniques have been employed in these comparisons. These will be discussed in context of the best practices and techniques that have produced the best rotor stability. Various factors which may affect rotor stability will also be discussed, and data taken at NIST as well as that found in the literature will be presented. The goal of this study is to establish the best practices for the use of a spinning rotor gauge as a transfer standard, as well as establishing the best stability that can be reasonable achieved.

10:40am **VT-MoM8 Pilot Study for International Comparison of Absolute Pressure Measurement from 3 × 10^{-9} Pa to 9 × 10^{-4} Pa**, ***Hajime Yoshida***, *K. Arai, E. Komatsu, K. Fujii*, National Institute of Advanced Industrial Science and Technology (AIST), Japan, *K. Jousten, T. Bock*, Physikalisch-Technische Bundesanstalt (PTB), Germany

International comparison of absolute pressure measurements in gas from 3x10^{-6} Pa to 9x10^{-4} Pa, identified as CCM.P-K3, was performed from 1998 to 2002 to determine the degree of equivalence of national metrology institutes (NMIs). A new international comparison, where the pressure range is expanded down to 3x10^{-9} Pa, is planned as CCM.P-K3 follower. A pilot study was performed in advance because two challenging issues are including the CCM.P-K3 follower. One is the stability of extreme high vacuum (XHV) gauge as a transfer standard. XHV gauges are kinds of hot cathode ionization gauges with a structure to reduce disturbances such as X-ray, electron stimulated desorption (ESD) ion, and so on. Few experiments are reported about the stability of XHV gauges from the viewpoint of metrology. The other is the stability of transfer gauges against to repeating bake-out because baking the calibration chamber including transfer gauges is inevitable to achieve XHV.

At first, three types of XHV gauges; Axial-symmetric transmission gauge (ATG), extractor gauge (EXG), and bent belt-beam gauge (3BG) were tested in NMIJ. Stabilities against air exposure and following bake-out were also tested for both XHV gauges and spinning rotor gauges (SRGs). A protocol for pilot study from 3x10^{-9} Pa to 9x10^{-4} Pa was prepared based on the results of both these tests and previous CCM.P-K3. ATG and EXG were adopted as transfer gauges because of achievements so far, although the stabilities of tested XHV gauges were comparable. In addition, two SRGs (SRG-1 and SRG-2) were also adopted as transfer gauges to calibrate at 9x10^{-4} Pa because stabilities of SRGs were expected to be better than those of XHV gauges. Measurement results of XHV gauges are normalized by those of SRGs. No significant differences of stabilities were observed whether the calibration gas was N_2 or Ar. N_2 was selected as the calibration gas because N_2 is typical calibration gas for ionization gauges.

A bilateral comparison between NMIJ and PTB was performed from May 2013 to Jan 2014 to confirm the effectiveness and to test the transport stability of the transfer standards. Shift of sensitivities of ATG, EXG, SRG-1, and SRG-2 were less than 0.58 %, 0.67 %, 3.5 %, and 0.28 %, respectively. The results of comparison were summarized except for data of SRG-1 because it clearly shows the drift of the sensitivity (effective accommodation coefficient). Results of the comparison show good agreement within the claimed uncertainty. Details will be presented at the conference.

11:00am **VT-MoM9 Stability of the Cold Cathode Ionization Gauge**, ***Paul Arnold***, *G.A. Brucker*, Granville-Phillips Vacuum Products

A study of the stability of cold cathode ionization gauges (CCIG) using an evaluation of physics principles affecting the ionization properties of the CCIG has been performed. The variation of the response to pressure of the CCIG is significantly dependent upon the interaction of the plasma discharge with the interior surfaces of the CCIG occurring during operation of the CCIG. New investigations will demonstrate these effects. The concept of measured pressure dose provides an index of likely drift in CCIG performance resultant from the magnitude of the plasma interaction, including the concept of remaining gauge useful life. The nature of the above plasma interaction changes the magnetic field internal to the CCIG as well as electron production from the cathode electrode, resulting in drift in CCIG performance. Both simulation and test data of these phenomena will be presented, showing relation of pressure dose to pressure performance with explanation of mechanisms.

11:20am **VT-MoM10 Cold Cathode Ionization Gauge Design Mitigates Well-known Performance Issues**, ***Brandon Kelly***, *G.A. Brucker*, Granville-Phillips Vacuum Products

Cold Cathode Ionization Gauges (CCIGs) are a well established indirect pressure measurement tool that has well documented advantages and shortcomings. In an attempt to overcome these design challenges a series of experiments have been conducted exploring different electrode geometries and configurations. Included in these experiments are high power magnet assemblies with unique discharge chamber geometries aimed at increasing device sensitivity at UHV pressures. Various modifications and methods have been tested to control the plasma discharge interaction with the interior of the CCIG for which increased lifetime will be shown. In addition to gauge sensitivity, other common shortcomings are addressed such as high vacuum starting time statistics and the separation of leakage current from plasma (i.e. discharge) current. A novel ionization chamber design prevents the escape of material from the internal surfaces of the gauge while preserving adequate conductance to the vacuum chamber and providing the ability to replace parts in the field. The theory and details of these new sensor design modifications will be discussed offering an insight into the next generation of cold cathode ionization gauges.

11:40am **VT-MoM11 A Systematic Study of Long-Term Vacuum Gauge Performance**, ***Gerardo Brucker***, *S. Heinbuch, T.C. Swinney*, Granville-Phillips Vacuum Products

Cold Cathode Ionization Gauges (CCIGs) have been the subject of numerous investigations throughout several decades. The free-running characteristics of their pure electron plasma, combined with the in-depth investigation of this phenomenon interacting with the interior of these devices, have led to numerous theories and speculation regarding the factors that regulate the long term performance of the gauges. Our laboratory has become increasingly interested in extending gauge lifetime through improved design and in the development of innovative dose calculation methodologies that can provide an adequate estimation of gauge remaining lifetime. We are particularly interested in understanding the phenomena that lead to drifts in gauge sensitivity with dose and specific gas chemistry. During our long term studies, gauge sensitivity is tracked over time against dose and the most modern surface and chemical analysis methodologies are employed to detect and understand the physico-chemical changes that take place at the internal electrodes. In this presentation we will demonstrate that while attempting to understand changes in gauge sensitivity it is equally important to consider changes in the magnetic as well as the internal electric characteristics of cold cathode gauges. Prior work by other groups has focused heavily on the influence of surface chemistry modifications. Our recent work will demonstrate that equal attention must be paid to the internal magnetic properties to fully account for long term changes in sensitivity.

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