Vacuum Technology
Room: 14 - Session VT-MoM

Vacuum Gauging and Metrology
Moderator: G.A. Brucker, Brooks Automation, Inc., Granville-Phillips Products

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Measuring the pressure inside sealed vacuum devices is a difficult proposition that typically requires destructive analysis. While commercial vacuum pressure gauges can be applied to large vacuum envelopes, the gauge's large volume and mass make them impractical for the small volumes of many vacuum devices. Incorporation of smaller volume pressure gauges such as the spinning rotor gauge or capacitance manometers can interfere with the device functionality. Measurements on these small vacuum devices can be done by a destructive technique where the device is punctured inside a calibrated ultra-high vacuum (UHV) vessel. With this technique the tube pressure is calculated from the changes in pressure of the UHV chamber and ratio of the chamber volume to tube volume. We have developed a non-destructive method by which the pressure inside high vacuum devices can be characterized without modifying or damaging the vacuum envelope. The approach transforms the existing vacuum device into a Penning or Redhead style ion gauge. We take advantage of the device features such as existing electrodes and high voltage standoff capability. By creating optimized crossed electrical and external magnetic fields around the vacuum envelope, we can generate a self-sustained Townsend discharge current which can be directly related to pressure. This technique is similar to the cold cathode gauge first developed by Penning, which was later modified by Redhead into the inverted magnetron gauge. In a typical cold cathode gauge the electrodes are a cylindrical design, with coaxial symmetry, which enables application of a uniform cross magnetic field. In many vacuum devices this coaxial electrode symmetry is not available. We overcome this obstacle by applying a ring-shaped magnetron magnet to the cathode electrode of a sealed vacuum device. In this arrangement the magnetic fields are not uniform, but the electron paths can still be significantly increased and even trapped resulting in enough ionization for a sustained discharge. The experimental technique to measure the time dependent pressure inside sealed vacuum devices non-destructively will be discussed. The novel concept of this technique is that we use the existing vacuum device as its own measurement gauge, with only the application of external fields and instrumentation. We have experimentally demonstrated a measurement range of 10-6 to 10-2 torr. A computer model of electron paths with some simple electrode geometries has been developed from which basic design guidelines can be derived. The model uses the Aleph finite-element particle-in-cell code developed at Sandia National Laboratories.

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

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

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