

Monday Morning, October 31, 2011

Vacuum Technology Division
Room: 111 - Session VT-MoM

Vacuum Measurement, Calibration & Primary Standards, Gas Flow and Permeation

Moderator: R. Garcia, SAES Getters

8:20am VT-MoM1 Transportable NIST Traceable Vacuum Standards for Secondary Calibration Laboratories and International Key Comparisons. *J.H. Hendricks, D.A. Olson, J.E. Ricker*, National Institute of Standards and Technology

Over the past decade, NIST has designed and built several high-stability transfer standard packages (TSPs) that have proven to be ideally suited for inter-laboratory comparisons in the atmospheric pressure and vacuum pressure range [1]. In the mid 1990's the development and use of micro electro mechanical systems (MEMS) enabled pressure sensor technology to make significant advances in both precision and accuracy. Resonant silicon gauges (RSGs) are MEMS sensors that are manufactured by micromachining silicon to produce silicon diaphragms nominally a few millimeters square by a fraction of a millimeter thick [2]. NIST has found that these gauges are very stable, rugged, and ideally suited as core technology for a high-stability precision pressure and vacuum standard. The RSG sensors with full-scale ranges of 10 kPa and 130 kPa have shown excellent stability [3]. However, one drawback of the RSGs is that they lack the sensitivity and resolution of capacitance diaphragm gauges (CDGs) with full-scale ranges of 133 Pa. The downside of only using these CDGs is their relatively poor calibration stability when compared to 10 kPa full scale range RSGs. The NIST solution has been to combine the high-resolution of the 133 Pa CDGs, with the high-stability of the 10 kPa RSGs into one transfer standard package. The RSG gauges are then used to determine the calibration drift in the CDGs at the time of use. A recently completed set of transportable NIST traceable vacuum standards with a range of 1 Pa to 10,000 Pa will be highlighted. These TSPs consist of pairs of 10 kPa RSGs and pairs of 133 Pa CDGs encased in temperature controlled enclosures that further enhance gauge performance. These standards will be used for an upcoming international key comparison in absolute pressure from 1 Pa to 10,000 Pa.

[1] Hendricks, J.H., Olson, D.A., Physics World, Vacuum Challenges and Solutions (2009) 18-19.

[2] Harada, K. et.al. 1999 Sensors and Actuators 73 261-266.

[3] Hendricks, J.H. et.al. Metrologia 44 (2007) 171-176.

8:40am VT-MoM2 Extending the Range of the Spinning Rotor Gauge for Vacuum Measurements and Calibrations. *M.L. Duncan, J.A. Keck*, Oak Ridge National Laboratory **INVITED**

The spinning rotor gauge (SRG) has long been the primary transfer standard used by metrology laboratories for providing traceability of measurements from field vacuum devices to primary vacuum standards maintained at national measurement institutes. The useful range of the SRG, in its current commercial form, is somewhat limited by several factors including the relatively small change in momentum of the rotating sphere caused by the adsorption/desorption of gas molecules as the gas pressure (and thus number of molecules adsorbing/desorbing) decreases. Current technology limits the low pressure end of this range to about $2E-5$ Pa with a $K=2$ uncertainty of approximately 1-3%.

Efforts are currently underway at the Oak Ridge National Laboratory to increase the range of the commercial SRG by approximately a decade by increasing the sensitivity of the rotor's momentum change to the low number of gas molecules available for adsorption/desorption at the lower pressures. Efforts are also underway to improve the uncertainty of the existing commercial SRG through better measurement and characterization of the thermal expansion of the spinning rotor itself. This paper will report on the progress of these efforts to date, some of the challenges discovered during the development process and plans to address those challenges

9:20am VT-MoM4 Cold Electron Source Used as Electron Source in Familiar Vacuum Measurement Devices. *P.C. Arnold*, Brooks Automation, Inc., *G.A. Brucker*, Brooks Automation, Inc., Granville-Phillips Products

Both a Bayard-Alpert type ionization gauge and a partial pressure analyzer have been fabricated and tested with cold electron sources. These vacuum measurement devices showed performance for their intended use generally similar to operation with thermionically heated hot cathode electron sources. The benefits from a cold cathode electron source are several:

reduced heat input to the system, lack of electron emission failure due to detrimental gases of the environment, non-susceptibility to deposits of the chemistry of the environment, non-interaction with the environment which interaction could produce gas species other than that occurring as part of the activity of the chamber, and finally fast turn-on to the electron emitting state. Details of the constructions, using an electron multiplier as the electron source, as well as the methods of controlling the electron emission current itself will be described. Test models, test conditions, and test results will be shown for a recently designed autoresonant ion trap mass spectrometer and an otherwise nearly traditional Bayard-Alpert ionization gauge.

9:40am VT-MoM5 Review of Thermal Conductivity Vacuum Gauges. *M. Wüest*, INFICON Ltd, Liechtenstein

Thermal conductivity gauges are ubiquitous in vacuum industry. In the form of Pirani gauges they are a mainstay of cost-effective measurement in the fine vacuum range. Different realizations of the Pirani gauge will be discussed from the classical heated wire sensor to the newer micromachined sensors. We will review not only basic parameters such as measurement range and sensitivity but also topics interesting for industrial applications such as dynamical response, robustness and inertness in industrial processes.

10:00am VT-MoM6 Investigation of the Hot Cathode Ionization Vacuum Gauge; Stability and Reliability on the Point of View of Traceability. *N. Takahashi*, ULVAC Inc., Japan

Many hot cathode ionization gauges have been developed focused on the lower limit of the pressure measurement. We have also developed the Axial symmetric transmission gauge (AT gauge) which lower limit of the pressure measurement is lower than 10^{-10} Pa.

On the contrast, stability and reliability are important for the industrial field and metrological traceability field.

Traceability of the vacuum gauge in the range of 1 to 10^{-4} Pa is established by the spinning rotor vacuum gauge. The uncertainty of the spinning rotor vacuum gauge includes its stability is estimated 1 % to few %. However, the sensitivity of the ionization gauges, which usually are the calibration item of the reference spinning rotor vacuum gauge, changes few % to few tenth %. Sensitivity change was caused by following; geometry change of the electrodes, change of the electron emission region on the hot cathode, contamination of the electrodes, etc.

We have investigated small all metal sealed triode type hot cathode ionization gauge on the contrast to the conventional glass bulbed triode type hot cathode vacuum gauge and all metal sealed BA gauge. The stability of the sensitivity of the small triode ionization gauge was less than 0.5% for 6 month before the huge earthquake happened in Japan. At the disaster, turbo molecular pump was crashed. We could report the sensitivity change of the gauge after the pump change.

We also demonstrate the benefit of the triode vacuum gauge in the contaminated vacuum system.

We will summarise the triode vacuum gauge is better characteristics than conventional glass bulbed triode vacuum gauge and BA gauge in the field of industrial field and metrological traceability field.

10:40am VT-MoM8 On the Stability of Capacitance-Diaphragm Gauges with Ceramic Membranes. *K. Jousten*, Physikalisch-Technische Bundesanstalt, Germany, *S.P. Naef*, INFICON Ltd, Liechtenstein

Capacitance-diaphragm gauges with ceramic membranes or diaphragms have been on the market for

about 15 years. The long-term stability of these devices with full scales from 13 Pa to 133 kPa

has been tested in the past decade by the calibration of gauges used by the manufacturer as reference

gauges on the production line. These reference gauges were calibrated annually on a primary

standard. It was found that the reproducibility of these devices depends on their full scale. For

13 Pa, the annual reproducibility near full scale varied between 0.02% and 0.05%, and for full scales

of 133 Pa and higher, it varied between 0.005% and 0.03% of full scale. The reproducibility of the

ceramic capacitance-diaphragm gauges for full scales of 133 Pa and 1.3 kPa was significantly lower

than the uncertainty of a primary standard applying the static-expansion method.

11:00am **VT-MoM9 Thermal Transpiration Effects in Capacitance Diaphragm Gauges with Helicoidal Baffle System**, *M. Vargas*, Institute of Mechanics - Bulgarian Academy of Sciences, *M. Wüest*, INFICON Ltd, Liechtenstein, *S.K. Stefanov*, Institute of Mechanics - Bulgarian Academy of Sciences

The Capacitance Diaphragm Gauge (CDG) is one of the most widely used vacuum gauges in low and middle vacuum ranges. This device consists basically of a very thin ceramic or metal diaphragm which forms one of the electrodes of a capacitor. The pressure is determined by measuring the variation in the capacitance due to the deflection of the diaphragm caused by the pressure difference established across the membrane. In order to minimize zero drift, some CDGs are operated keeping the sensor at a higher temperature. This difference in the temperature between the sensor and the vacuum chamber makes the behavior of the gauge to be non-linear due to thermal transpiration effects. This effect becomes more significant when we move from the transitional flow to the free molecular regime ($Kn > 0.1$). Besides, CDGs may incorporate different baffle systems to avoid the condensation on the membrane or its contamination.

In this work, the thermal transpiration effect on the behavior of a rarefied gas and on the measurements in a CDG with a helicoidal baffle system is investigated by using the Direct Simulation Monte Carlo method (DSMC). This technique is based on the discretization of the number of particles, the space and the time domains, and it combines deterministic aspects for modelling the particle motion with statistical aspects for computing the collisions between particles. The study covers the behavior of the system under the whole range of rarefaction, from the continuum ($Kn < 0.01$) up to the free molecular limit ($Kn > 100$), for various temperature differences and different temperature gradient configurations (with radial and axial components). Moreover, in order to analyse the dynamic response of the system to a change in the sensor temperature from an initial isothermal configuration, some non-steady state calculations are performed. In this way the evolution of the macroscopic properties of the gas is studied from the initial moments until the steady state is achieved.

11:20am **VT-MoM10 Direct Conductance Measurements of Laser-Drilled Pinhole Apertures**, *J.A. Fedchak*, *D.R. Defibaugh*, National Institute of Standards and Technology

A pinhole orifice with a known conductance can be used as a secondary flow standard. We are interested in using pinhole orifices to produce nitrogen gas flows into vacuum in the range of 10^{-11} mol/s to 10^{-6} mol/s (10^{-7} to 10^{-2} cm³/s; STP) for vacuum gauge calibrations because a flowmeter based upon an appropriate set of orifices is easy to operate and automate. Commercially available laser-drilled pinhole orifices with diameters from 1 μ m to 50 μ m can have molecular-flow conductances, C_0 , ranging from about 0.1 μ L/s to 230 μ L/s for N₂ at 23 °C, and can be used to produce gas flows in the range of interest by applying an upstream pressure in the range of 10 Pa to 100 kPa (0.1 to 760 torr). Accurate measurements of the orifice conductance, C , as a function of pressure are required to use the pinhole orifice as a basis of a flowmeter. The NIST bellows flowmeter is a primary gas flow standard that was used to directly measure the conductance of a pinhole orifice to better than 0.2 % over the entire pressure range of interest. We present results of the conductance measurements for nitrogen and other gases. One might expect that the differences among the gases would be mainly due to their different thermal velocities, and that those differences would disappear when the normalized reduced flow rate (C/C_0) is plotted as a function of inverse Knudsen number. However, this was not the case at higher pressures. For example, the reduced flow rate for nitrogen was smaller than for argon at the same inverse Knudsen number. Following a suggestion by Jitschen (Vacuum 76 (2004) 89-100), the effect of the heat capacity ratio on C/C_0 was investigated.

Authors Index

Bold page numbers indicate the presenter

— **A** —

Arnold, P.C.: VT-MoM4, **1**

— **B** —

Brucker, G.A.: VT-MoM4, **1**

— **D** —

Defibaugh, D.R.: VT-MoM10, **2**

Duncan, M.L.: VT-MoM2, **1**

— **F** —

Fedchak, J.A.: VT-MoM10, **2**

— **H** —

Hendricks, J.H.: VT-MoM1, **1**

— **J** —

Jousten, K.: VT-MoM8, **1**

— **K** —

Keck, J.A.: VT-MoM2, **1**

— **N** —

Naef, S.P.: VT-MoM8, **1**

— **O** —

Olson, D.A.: VT-MoM1, **1**

— **R** —

Ricker, J.E.: VT-MoM1, **1**

— **S** —

Stefanov, S.K.: VT-MoM9, **2**

— **T** —

Takahashi, N.: VT-MoM6, **1**

— **V** —

Vargas, M.: VT-MoM9, **2**

— **W** —

Wüest, M.: VT-MoM5, **1**; VT-MoM9, **2**