Monday Afternoon, October 29, 2012

Vacuum Technology
Room: 14 - Session VT-MoA

Gas Flow, Leaks, Permeation and Mass Analysis
Moderator: J.A. Fedchak, National Institute of Standards and Technology

2:00pm **VT-MoA1 First International Comparison of Standard Leak Calibrations of Metrological Institutes**, ***K. Jousten***, PTB, Germany, *K. Arai*, NMIJ, Japan, *U. Becker*, *O. Bodnar*, PTB, Germany, *F. Boineau*, LNE, France, *J.A. Fedchak*, NIST, *V. Gorobey*, VNIIM, Russian Federation, *W. Jian*, SPRING, Singapore, *D. Mari*, INRIM, Italy, *P. Mohan*, NPL/I, India, *J. Šetina*, IMT, Slovenia, *B. Toman*, NIST, *M. Vicar*, CMI, Czech Republic, *YH. Yan*, NIM, China **INVITED**

The measurement of leak rates has become an important test in industry for function tests, quality and safety management, and for environmental protection. Leak tests are performed not only for vacuum chambers, but also for various containers like electrical high power switches, pace makers, refrigerating systems, isolation vacuum, rims, and tanks. The leak tests are performed by leak detectors which mainly use helium as test gas. Traceability to the SI units is given by calibrated standard leaks that emit a well known flow rate of typically helium gas. Many National Metrological Institutes (NMIs) provide such traceability in their vacuum sections.The NMIs that signed the mutual recognition arrangement committed themselves to prove equivalence of their calibration measurement capabilities. The test of equivalence is a comparison where a transfer standard is calibrated at the participants' facilities and the results compared. The difference between the laboratory result and a reference value or a bias of a laboratory must not exceed the uncertainty of this difference or the bias.To test equivalence of standard leak calibrations NMIs from 11 countries performed a comparison with two helium permeation leaks as transfer standards. The leak rates were 4E-11 mol/s for standard leak L1 (1E-4 Pa L/s at 23°C) and 8E-14 mol/s (2E-7 Pa L/s at 23°C) for L2 respectively. For the latter, only 6 NMIs had measurement capabilities. Since leak rates from permeation leaks decrease with time, special evaluation procedures had to be applied to calculate a reference value and to compare the results of the NMIs. Also a statistical method was applied to evaluate a possible bias of a laboratory. Most of the 11 laboratories proved equivalent in the case of transfer standard L1 and all for L2. These results will be published and serve as basis for mutual recognition of calibration results.

2:40pm **VT-MoA3 Comparison of the Flow Ratio of the Permeation Type Helium Standard Leaks and the Gas Flow Generator Composed with a Small Conductance Element Made by Sintered Stainless Steel**, ***N. Takahashi***, ULVAC Inc., Japan, *H. Yoshida*, AIST Japan

The possibility of the gas generator with a small conductance element made by sintered stainless steel as a small gas flow standard is examined.

Helium leak test is one of an important way for the non-destructive testing. Helium standard leaks are usually used for the calibration and for the adjustment of the sensitivity of helium leak detectors. Characteristic and calibration of the helium standard leaks were well studied from late 1980's to middle 1990's1-6). However, the flow ratio of the one unit of standard leak is fixed. Helium flow ratio by the permeation through glass element usually shows large temperature dependence.

On the other hand, the gas flow generator composed with a small conductance element made by sintered stainless steel was reported as a variable, stable and wide range gas flow generator for the vacuum gauge calibration7). We examined and made comparison of the helium gas flow ratio between the gas generator and the permeation type helium standard leaks. The flow ratios of the permeation type helium standard leaks are traceable to AIST/NMIJ. The relation of the helium gas flow generated by the generator and the permeation leaks was good agreement with less than 5% of difference. The generator generates wide range of helium gas flow less than 10-5 Pam3/s. This experiment was performed on the ultra high vacuum equipment, which residual pressure was lower than 10-7 Pa, with a quadrupole mass spectrometer. The generator is also useful for the wide range, stable and easy way of calibration of standard leaks. Stability, conformity of the generator is also reported.

1) L. E. Bergquist, et al., JVST **A 7**, 1989, 2414.

2) M. V. Iverson, et al., JVST **20**, 1982, 982.

3) J. M. Ball, JVST **A 6**, 1988, 2860.

4) W. G. Bley, Vacuum **41**, 1990, 1863.

5) P. J. Abbott, et al., JVST **A 14**,1996, 1242.

6) C. D. Ehrlich, et al., JVST **A 10**, 1992, 1.

7) H. Yoshida, et al., Vacuum **86**, 2012, 838.

3:00pm **VT-MoA4 Porous Plug Made of Sintered Stainless Steel used as Standard Conductance Element**, ***H. Yoshida***, *K. Arai*, *T. Kobata*, National Institute of AIST, Japan

A porous plug made of sintered stainless steel, which is named as standard conductance element (SCE), has been developed as an open-type standard leak element for in-situ calibration of ionization gauges (IGs) and partial pressure analyzers [1]. Since the pore size of sintered filter is less than 1 micro meter, the molecular flow condition is realized up to 10 kPa of the upstream pressure of SCE. Therefore, four useful characteristics shown in below are available. (1) Flow rate is proportional to the upstream pressure of SCE. (2) Introducing various gas species with known flow rate is available by applying this single leak element. (3) Calibration using mixture gases is available. (4) Dependence of flow rate on the temperature is small and easy to compensate. In addition, the molecular conductance C_S of SCE has good long-term stability of less than 3 %/year typically. No significant influence is observed by introducing water vapor and bake-out. Since SCE with C_S from $1x10^{-10}$ m^3/s to $2x10^{-9}$ m^3/s is available, the flow rate of less than around 10^{-5} Pa m^3/s is generated with various gas species.

Three typical applications of SCE are introduced. First is the quantization of the gas desorption rate for thermal desorption spectroscopy (TDS) [2]. The gas desorption rate was measured by a quadrupole mass spectrometer (QMS) which was calibrated by SCE with H_2, H_2O, N_2, CO, and CO_2 gases. Second is the measurement of the effective pumping speed S_{eff} of cryopump in the range from 10^{-9} Pa to 10^{-6} Pa [3]. H_2, CH_4, N_2, and Ar gases with known flow rate Q were introduced into the test chamber with the cryopump through SCE, and measured the pressure increment p by extreme high vacuum gauges. Pressure indications of the gauges were compensated by relative sensitivity factors for N_2. The last one is the in-situ calibration of IG and QMS. The standard pressure p was obtained from Q divided by S_{eff}, where Q and S_{eff} were determined by using SCE and conductance modulation method, respectively. The sensitivity of IG and QMS were measured for H_2, He, CH_4, H_2O, Ne, CO, N_2, C_2H_4, C_2H_6, O_2, Ar, C_3H_6, CO_2, N_2O, and C_3H_8.

These results show that SCE seems to satisfy almost all of requirements for the quantitative measurements in high and ultrahigh vacuum. SCE will be used as a new vacuum standard device.

[1] H. Yoshida, et.al., Vacuum 86 (2012) 838-842.

[2] S. Inayoshi, et.al, 52th Annual Symposium of the Vacuum Society of Japan (AVSSJ-52), Tokyo, Japan, 2011.

[3] M. Yamamoto, et.al., Proceedings of international particle accelerator conference (IPAC2011), San Sebastian, Spain, 2011, 979-981.

3:40pm **VT-MoA6 Measurement of Gas Transport in Solids by a Saturation/Outgassing Method**, ***L. Wang***, *J.A. Tanski*, *R.Y. Weinberg*, Los Alamos National Laboratory

Common flow techniques for measuring gas transport properties in solids requires the formation of a membrane which serves as a barrier to the movement of gas and the formation of leak-tight seal so the gas is diffusing through the membrane. However, for many materials, especially brittle non-metallic ionic solids, meeting these two requirements are difficult. In this study, an alternative method based on first saturating the material with the gas at a known pressure and subsequently allowing the absorbed gas to diffuse out to a static vacuum environment was developed for measuring gas transport properties of this type of materials. The method uses a sample of a well-defined geometry, in this case a right cylinder, and a computer program (DiSol) was developed to model the desorption process and extract diffusivity and solubility from the measured outgassing curve. To demonstrate the viability of the method, helium transport in a high-density polyethylene (HDPE) cylinder was measured, and the results obtained compared well with the published diffusivity and solubility values. The details of the method and the HDPE data obtained in this study will be presented.

4:00pm **VT-MoA7 Report on Workshop on Measurement Characteristics and Use of Quadrupole Mass Spectrometers for Vacuum Applications**, *K. Jousten*, Physikalisch Technische Bundesanstalt, Germany, *J. Šetina*, Institute of Metals and Technology, Slovenia, ***R. Ellefson***, REVac Consulting

The ISO Technical Committee (TC) 112 is responsible for international standards in the field of vacuum technology. Supporting TC112 are three working groups (WG): WG1 is responsible for vacuum pumps, WG 2 for vacuum instrumentation and WG 3 for vacuum hardware. Hence, the WG 2 is responsible for total and partial pressure measurement in vacuum. In 2006, WG 2 began a project towards standardization for the specification and calibration of quadrupole mass spectrometers (QMS). A new ISO (Draft) standard 14291 "Definitions and specifications for quadrupole mass spectrometers" will be published in 2012. Additionally WG 2 is working on documenting the proper use of a QMS and establish a standard for meaningful calibration procedures. In support of this ISO goal, the European Metrology Research Programme has established a project IND12 "Vacuum metrology for production environments" to open new measurement capabilities for vacuum and to help industry to characterize vacuum in industrial environments.

One of the focuses of this IND12 effort is to address traceability to national measurement standards for partial pressure measurements and outgassing rate measurements for materials characterization in industry. To gather current status on such methods, a workshop on calibration of the QMS for industrial use was held in Bled, Slovenia. Quadrupole mass spectrometers are widely used to measure partial pressures in vacuum although the performance and accuracy of these measurements is the subject of ongoing discussion among users. The wide range of partial pressure measurement needed from UHV/XHV (10^{-10} Pa) to 1 Pa emphasizes that one type of QMS does not address all applications. From presentations at this workshop, application-specific data on performance of QMS types gives guidance for selection of a QMS type for an application. Talks were given on setup and ion source operation recommendations; initial calibration methods for an application and *in situ* calibration methods for verification or recalibration of a QMS during use. The fact that QMS electron energies differ by application and manufacturer leads to the requirement that gas species calibration is needed for each QMS for accurate partial pressure or compositional analysis. Papers on the electron-ion space charge at pressures>10^{-4} Pa in ion sources were given together with gas interference where addition of a partial pressure of a new chemical species B gives a different ion current output for species A (when A is known to be at its original partial pressure). Results of these talks at the workshop will be summarized in this presentation.

4:20pm **VT-MoA8 Mass Spectrometry a Mile Deep: Issues and Solutions for Underwater Vacuum Systems**, ***R.T. Short***, SRI International **INVITED**

There are numerous advantages to performing in situ chemical analyses in the field rather than collecting samples for laboratory analysis. These include reducing the possibility for sample contamination or degradation, significantly improving the spatial and temporal resolution of analyses, and providing the ability to implement adaptive sampling strategies by receiving the analytical information in real-time or near real-time. Many analytical instruments, such as mass spectrometers (MSs), require high vacuum for operation. Vacuum systems for these portable instruments must be reasonably small, low-power, and often more rugged than needed for laboratory operation. Underwater instruments of this type have the additional requirements that the vacuum systems must be self-contained (or exhaust to a high-pressure environment) and the sampling interfaces must withstand pressure differentials greater than 1 atmosphere – hydrostatic pressure increases by approximately 1 bar for every 10 m depth in the ocean. Consequently, all of these constraints must be taken into account when selecting or building vacuum systems for these applications.

Several groups around the world have been working on developing and using underwater MSs for in situ chemical measurements in oceans, lakes, and rivers – at times over a mile deep. To date, all of these operational underwater MSs have employed membrane-inlet interfaces to introduce analytes into the vacuum of the MS. Analytes that are soluble in the membrane material (typically polydimethlysiloxane or Teflon) permeate through the membrane and evaporate into the ion source of the MS, where they are ionized by an electron impact source. For operation at depth, the membrane must be supported by a porous frit to withstand the increased hydrostatic pressure. Membrane inlet MSs are very effective at detecting and quantifying dissolved gases, light hydrocarbons, and volatile organic compounds, often at trace levels.

Design considerations for underwater MSs will be discussed in this presentation, along with examples of specific components chosen for the vacuum systems. In particular, the focus will be on problems with operating vacuum systems under these often extreme and harsh conditions, along with solutions to mitigate these problems. In addition, we will present examples of deployments of underwater MSs for a variety of applications on a number of unmanned deployment platforms.

5:00pm **VT-MoA10 Performance Optimization for Autoresonant Ion Trap Mass Spectrometers**, ***G.A. Brucker***, *J. Rathbone*, *B. Horvath*, Brooks Automation, Inc., Granville-Phillips Products

A new generation of residual gas analyzers based on autoresonant ion trap mass spectrometry (ARTMS) technology has recently become commercially available and is rapidly gaining market acceptance for vacuum processing applications. Recent investigations into this novel ion trap technology have focused on a detailed understanding of the role of ion generation and storage on the mass spectrometer's performance. The rate of ion formation, the initial energetics of the ions stored inside the trap, the shape of the electrostatic trapping potential and the characteristics of the radiofrequency signal sweep used to eject the ions all have a significant impact on the performance of an ART MS instrument. Unit-to-unit variations in ion formation rates and energetics due to mechanical assembly tolerances, can lead to slight variations in ion trap performance which can be easily corrected through adjustment of the electron beam focusing optics and the shape of the electrostatic trapping potential. This presentation describes a straight forward methodology that has been developed in our research laboratory to manually optimize ion trap performance during routine measurements and also an automated tuning procedure (Auto-Tune) that is built into the control software and can be used to restore trap performance each time an ART MS sensor is fitted with a new filament assembly.

5:20pm **VT-MoA11 Differential Pumping Method for a Fast Partial Pressure Analyzer of Recent Design to Extend its Upper Pressure Limit Up to the Torr Range**, ***P.C. Arnold***, *T.C. Swinney*, Brooks Automation, Inc., Granville-Phillips Products

The upper pressure limit of a partial pressure analyzer (PPA) was extended by differential pumping so that gas composition of process pressures are evaluated. The selection of orifice size for differential pumping, first by calculation, then by empirical data, is presented. The range of a linear relation for reduced total pressure vs. process pressures in the millitorr range for several gases is presented. The validity for scans of multiple gas species for 0 – 130 amu was also evaluated by testing, comparing scans in the high pressure region to those in the reduced pressure region. The application of this method for an autoresonant ion trap will be presented for the first time in this pressure reduction protocol as it provides measurement of fast transients at these higher pressures. Trade-offs for orifice sizes and pressure reduction ratios will be discussed. The test set-up as it relates to the AVS Recommended Practices and the conclusions for pressure ranges that it provides will be given.

Authors Index

Bold page numbers indicate the presenter