Vacuum Technology Division
Room: 111 - Session VT-MoA

Optical and Mass Spectroscopy for Gas Analysis and Pump Modeling

Moderator: R. Versluis, TNO Science and Industry, The Netherlands

2:00pm **VT-MoA1 Low Uncertainty Measurements of Trace Water Vapor Based on Cavity Ring-Down Spectroscopy**, ***T. Hodges***, National Institute of Standards and Technology **INVITED**

I will discuss how cavity ring-down spectroscopy (CRDS) can be applied to accurately measure the concentration of residual water vapor which is present in a vacuum system or process gas stream. In CRDS, a monochromatic laser beam is injected into an evacuated or sample-gas-containing optical resonator and the transient decay of light exiting the cavity is monitored to quantify the optical losses. For water detection, the laser wavelength is tuned to probe characteristic rotation-vibration absorption features of the water molecule. The sample absorption coefficient is determined from observations of the ring-down cavity decay time and laser frequency, both of which can be precisely measured. Also, because CRDS uses a resonant optical cavity, extremely long effective optical pathlengths (up to tens of km) can readily be achieved in the laboratory. These properties make CRDS a high-spectral resolution, species-selective method, with relatively small combined uncertainty, and high sensitivity. I will show that when CRDS measurements are combined with first-principles spectroscopic models, this technique can yield concentration measurements with sub-percent-level relative uncertainty for absolute concentrations as low as 10^{11} cm^{-3}. I will present examples of CRDS-based trace water detection in vacuum and ultra-high purity gas systems, and I will talk about recent CRDS measurements of the vapor pressure of ice over the temperature range 0 deg C to -100 deg C.

2:40pm **VT-MoA3 Comparison of Cavity Ring-Down Spectroscopy, Oscillating Quartz Crystal and Electrical Impedance Technologies for Trace Water Vapor Detection below 100 ppb**, ***M.W. Raynor***, *J. Feng*, Matheson

Control of trace water vapor in high purity process gases at low ppbv levels is critical to the performance of many micro-electronic and photonic devices [1]. Consequently a variety of measurement technologies, have been developed to detect water below 100 ppb. However, the performance characteristics of each technology can vary and this is not always well understood by users. In this presentation three different approaches are considered: Oscillating quartz crystal microbalance (QCM), Al_2O_3 based electrical impedance sensor and laser induced cavity ring-down spectroscopy (CRDS). QCM technology, developed in the early 1960's, is still widely applied today. It is based on adsorption of water vapor on the hygroscopic coating of the QCM, which causes an increase in the mass of the crystal, and in turn, decreases its oscillation frequency. CRDS is a laser absorption technique based on the light decay in a high finesse optical cavity. The high resolution laser ~1-2 MHz (~10^{-5} cm^{-1}), high reflectivity mirrors (~ 0.99998) results in a long effective path-length which enables high selectivity and sensitivity for H_2O detection. Impedance-based sensors for trace water vapor detection have typically suffered from drift and equilibration issues. However, recently an impedance-based Al_2O_3 sensor chip with integrated heater for cycling the temperature within 60ºC to 200ºC has been developed. Water vapor is measured dynamically as impedance changes during wet-up of the sensor resulting in rapid response. In this work, we present and discuss data showing the performance of the above detection technologies with respect to sensitivity, speed of response and measurement stability in the <100 ppbv range.

[1] H.H. Funke et al., Rev. Sci. Instrum., 74 (9) 2003, 3909-3933.

3:00pm **VT-MoA4 Commercial Applications and Benefits of Continuous-Wave Cavity Ring-Down Spectroscopy**, ***Y. Chen***, Tiger Optics

Continuous-Wave Cavity Ring-Down Spectroscopy (CW-CRDS) is a laser-based state-of-the-art detection technique. Based on first principles, it directly derives the absolute optical loss due to absorption inside the cavity from a simple time measurement, independent of laser intensity noise and optical detector drift.

This highly sensitive absolute absorption measurement technique was first commercialized by Tiger Optics for sub parts-per-billion (ppb) level detection of moisture in inert gases. More than a dozen national metrology labs now use this technique as their moisture transfer standard. Several of these key national labs recently concluded a multi-year project comparing the performance of their different moisture standard generators by using two Tiger Optics CW-CRDS devices as their "referees", shipping them around the globe across three different continents. Inter-comparison data from this comprehensive study will be presented.

With over 1000 measurement points worldwide, CW-CRDS has gained widespread acceptance and growing use in a series of challenging, real-world industrial applications, well beyond trace moisture in simple matrices. We will demonstrate the strong capability of CW-CRDS for a diverse group of analytes over a large dynamic range and under widely varying application conditions. In addition, starting with ultra-high-purity, sub or low parts-per-billion measurement, this technology is now increasingly sought for higher parts-per-million applications as well. Taking maximum advantage of its exceptional dynamic range, with a variety of flexible configurations, CW-CRDS-based instruments address these various applications with a self-verifying measurement solution that is fast and sensitive, yet extremely robust and simple to operate.

3:40pm **VT-MoA6 Vacuum Quality Measurement at UHV Levels with AutoResonant Ion Trap Mass Spectrometers**, ***G.A. Brucker***, Brooks Automation, Inc., *J. Rathbone*, *B.J. Horvath*, Brooks Automation, Inc., Granville-Phillips Products

Autoresonant Ion Trap Mass Spectrometers (ART MS) have recently become commercially available and are rapidly finding applications in many areas of the vacuum technology industry. One of the biggest benefits of ART MS sensors is their ability to provide fast and sensitive data at ultrahigh vacuum levels (UHV). The ability to operate the sensor remotely, i.e. with the electronics unit away from the gauge head, has also made ART MS technology the gas analysis instrumentation of choice for hard radiation environments. The performance of ART MS sensors under UHV conditions is discussed. Test results for gas analysis measurements performed under UHV conditions are shown and compared against similar results obtained with legacy instrumentation including quadrupole-based residual gas analyzers. Different approaches available to improve the performance of an ART MS instrument under UHV conditions are explored and explained based on the basic principles of operation of the technology.

4:00pm **VT-MoA7 Reducing Uncertainties for Hydrogen Loading Determination of 1,4-bis(phenethyl)benzene (DEB) Using GC/MS Instead of the Traditionally-used CHN Analysis Method**, ***S.M. Thornberg***, *J.M. Hochrein*, *M.I. White*, Sandia National Laboratories

Hydrogen getters are used in many industries including aerospace, defense, and electronics (e.g., MEMS packaging) to control levels of hydrogen in sealed atmospheres and vacuum systems. In this research, we explore not only the products formed during the hydrogenation process but also the product distribution resulting from differing rates of hydrogenation. This work focuses on the analysis of hydrogenation products of 1,4-bis(phenethyl)benzene (DEB) using GC/MS and a method for creating samples with known hydrogenation levels. This information can then be used to calculate the remaining capacity of the getter by determining the ratios of saturated, partially saturated, and unsaturated products.

DEB has a molecular formula of C22H14 (MW=278 amu) and has a capacity for four moles molecular hydrogen per mole of DEB. The analysis of pure DEB (unhydrogenated) showed no contamination from partially hydrogenated products and only one chromatographic peak (from DEB). As hydrogenation proceeds, a series of products is formed with nominal masses 280, 282, 284, and 286 amu (282, 286, 290 and 294 amu for deuterium). Hydrogenation experiments were performed from 0% to 100% hydrogenation (calculated by molar ratios) with hydrogen mixed with a buffer gas (nitrogen) to slow the uptake reaction rate. After hydrogenation, the resultant solid was homogenized, dissolved in methylene chloride, and filtered. The stock solutions, diluted appropriately, were then analyzed using gas chromatography (Agilent, model 6890N) for product separation, and a high-resolution mass spectrometer (Jeol MStation, model JMS-700) for product identification.

In this talk, a comparison between the GC/MS method presented here and the traditional CHN analysis method will be presented. Round robin samples between three labs were used to assess the performance of each method.

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin company, for the U.S. Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

4:20pm **VT-MoA8 Sampling Equilibration Times of Chemical Species for Different Capillary Surfaces**, ***R. Ellefson***, REVac Consulting, *D. McClelland*, Mound Technical Solutions, Inc.

Gas sampling through long, small-bore capillary tubing has long been used as a method to reduce the atmospheric pressure (or higher pressure) process gas to a low pressure for analysis by a mass spectrometer (MS). With care of sampling system design and operation, the species integrity of the process gas can be preserved in the sampling which enables accurate compositional analysis. The gas dynamics of the gas stream within the capillary tube equilibrating with the capillary wall and measured at the MS leads to a stable composition at the MS when equilibrium is achieved. A model that includes the wall material interaction is presented with prediction of stabilization time for various gas species. Data from four different capillary materials or surfaces are given to show the interaction process. The capillaries tested are: 304SS, 304 Sulfinert® SS, PEEK (polymer) and fused silica tubing. All capillaries have 0.25 mm i.d. and a 2 m length for direct comparisons. Composition profiles versus time are measured for a dry nitrogen sample followed by room air (50% RH) which shows the gas dynamics of the equilibration of adsorbed gases (e.g. H_2O and CO_2)) with the various interior surfaces of each capillary. The effect of capillary length and i.d (defining the surface area to be equilibrated) is included in our model and measurements. Equilibration times of 50 sec for H_2O are seen at room temperature for a 2 m capillary with 10 sccm flow rate. Longer times are needed to reach the low H_2O concentration in the nitrogen (drying the capillary surface). Raising the temperature of the capillary reduces equilibration time as expected.

The exit end of the capillary flows into the low pressure region created by the sampling forevacuum with a port to the MS for analysis. The effect on equilibration time of a Silcotek® surface treatment of interior surfaces of the inlet to the MS is measured and compared with equilibration time for the regular 304SS surface of the machined inlet.

4:40pm **VT-MoA9 Numerical Methods for the Design of Vacuum Systems with Examples**, ***R. Kersevan***, ITER International Organization, France **INVITED**

The paper deals with the issue of the numerical computation of relevant properties of vacuum systems under ultra-high vacuum (UHV) conditions, i.e. when molecular flow conditions are in place. Properties of interest, among others, are pressure profile, angular profiles, conductances, transmission probabilities, effective pumping speed, sputtering deposition profiles.

Many modern research tools need UHV conditions in order to function properly. The size of the system is not an issue, it can be very small (electronic packaging; gauge calibration benches, for instance) or very large (ITER torus, cryostat and ancillary systems; particle accelerators; spectrometers, etc...). The availability of relatively cheap computing power has in recent years brought at the forefront of research new software tools which allow the simulation of complex geometries and working conditions.

The paper quickly reviews the existing algorithms and tools [1], and then moves on to show examples of calculations, with particular emphasis on the Molflow+ code [2].

[1] R. Kersevan, "Analytical & Numerical Tools for Vacuum Systems", Proc. CAS - CERN Accelerator School and ALBA Synchrotron Light Facility : Course on Vacuum in Accelerators, Platja d'Aro, Spain, 2006 - Downloadable at http://cdsweb.cern.ch/record/923393

[2] R. Kersevan, J-L. Pons, "Introduction to MOLFLOW+: New graphical processing unit-based Monte Carlo code for simulating molecular flows and for calculating angular coefficients in the compute unified device architecture environment", J. Vac. Sci. Technol. A 27, 1017 (2009);

5:20pm **VT-MoA11 Numerical Modeling of Compact Siegbahn Molecular Drag Stages**, *H. Telib*, Politecnico di Torino, Italy, *R. Arpa*, Optimad Engineering s.r.l., Italy, *L. Campagna*, ***I.F. Cozza***, *E. Emelli*, Agilent Technologies s.p.a., Italy

In the frame of an optimization of single/multi-stage disk-type vacuum pumps, characterized by spiral channels a comprehensive but efficient numerical analysis of performances has to be founded on a careful modeling of the local gas flow features, such as pump leakage and development of the rarefied gas flow along the curved channels. Here, gas flows are in general considered three-dimensional, because of the spiral groove curvature, and driven by pressure gradients and the applied rotation speed as well as inertial forces (centripetal and Coriolis effects), which play the most important role.

Following the assumptions made for a Holweck model by Sharipov et al., we propose a lower-order model for steady flows in spiral molecular drag stages, based on the solution of the Boltzmann Equation (BE) with a BGK closure, in general curvilinear coordinates (properly fitted to the geometrical design of the channel), where the inertial effects explicitly appear in the governing equation. The order of the 3D original problem is reduced in the physical space (2D), by introducing assumption of “locally” known flow development of the distribution function along the spiral channel. Thus, 2D-BE calculations of the flow rates and stresses will be performed in a finite number of sections, suitably positioned along the spiral channel, from the outlet up to the inlet, in order to recover the pressure and torque distribution. In particular, the 2D Boltzmann equation is linearized in the most significant parameters (local rotation speed and pressure gradients along the pump radial direction), and solved in the reference section. The local valus of pressure and torque are obtained consistently by enforcing the mass flow conservation constraint.

A Discrete Velocity Method (DVM) is used to solve the Boltzmann Equation, with an explicit pseudo-time dependent technique to relax the flow up to its stationary solution. In order to decrease the computational time employed, the solver is designed to work on parallel architectures (MPI).

The performance prediction of the model will be assessed using test cases from the literature and compared to the available experimental data, on both Holweck and Siegbahn geometries. A further verification test will be carried out, to test prediction capabilities in the continuum regime by direct comparison with results obtained by a Navier-Stokes solver, with slip-boundary conditions.

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