

# Monday Afternoon, November 9, 2009

## Vacuum Technology

Room: C1 - Session VT-MoA

### Pressure, Partial Pressure, and Flow Measurement

Moderator: J. Setina, IMT

#### 2:00pm VT-MoA1 Investigations on the Dynamic Response of Pirani Gauges, *M. Wüest, B. Andraeus, R. Stocker*, INFICON, Liechtenstein

For over 100 years the Pirani sensors measure vacuum pressure from  $\sim 5 \times 10^{-5}$  mbar to atmosphere. Pirani sensors are based on heat conduction through gas. Due to this measurement principle the accuracy of Pirani sensors is reduced near atmospheric pressure. Yet many industrial processes use the low cost Pirani sensors for venting applications and the trend is to faster cycle times. Depending on the construction Pirani sensors can display various inaccuracies during rapid pressure changes. This is because the wall temperature measurement used for temperature compensation lags the pressure change. Here we present an investigation to optimize Pirani sensor design for rapid pressure venting applications.

#### 2:20pm VT-MoA2 Portable Gas Sampling Instrument Capable of Measuring Leak Rates, Volumes, and Pressures without A/C Power, *S. Thornberg, J. Brown*, Sandia National Laboratories, *L. Miller, J. Ithaca*, B&W Pantex

Certain operations (e.g., volume measurement, gas sampling, and leak rate determination) commonly employed in the field of vacuum technology typically require A/C-powered equipment (vacuum pumps, electrometers, computers, etc.) to perform the measurement or operation. However, some hazardous applications require the cessation of A/C-powered operations when certain conditions exist like the presence of a thunderstorm that can generate dangerous voltage spikes from nearby lightning strikes. To alleviate this problem, a new instrument designed and prototyped at Sandia National Laboratories is capable of performing leak tests (greater than  $5 \times 10^{-3}$  atm cc/s air), measuring the internal volumes of complex manifolds (up to liters in volume), making absolute pressure measurements, and performing gas sampling, all without the use of A/C power. The system is designed to be very easy to use with many pneumatic valves behind the instrument panel that are controlled by simple control valves specifically designed/invented for this application. Other functions this instrument can perform are the generation of a modest vacuum (approximately 10 Torr) and backfilling the system with a user-supplied gas. The system has been prototyped, and production models have been fabricated and are in use on production lines. This presentation will highlight the design and features that enable these operations to be performed without A/C power in an operator-friendly package that is not much larger than a thick briefcase. Extensive qualification testing has been performed using these instruments, and the results will be presented to show the performance and NIST traceability. (Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.)

#### 2:40pm VT-MoA3 Mass Spectrometer Sampling of Transient Gas Compositions in Processes, *R.E. Ellefson*, Consultant

Many modern vacuum processes involve transient gas densities and compositions. Analysis of the composition of process gas requires the transport of a sample from near the process point of interest to the gas analyzer. The transport time and surface conditioning time of the sampling apparatus needs to be much shorter than the cycle times of the process to give useful composition-versus-time profiles. Many processes for CVD or ALD deposition involve metal organic precursor gases that adsorb on surfaces conformally as part of the deposition process. These gases also adsorb on the sampling system internal surfaces. Management of the surface coverage dynamics for each process is an important factor for good sampling. In this paper, the time constants for species diffusion within the process, sample gas transport and surface coverage times for adsorption and desorption as the composition changes are defined and calculated for typical applications. Strategies for accelerating the stabilization of the sampling system surface composition by controlling surface temperature in relation to the process temperature are discussed. Designs and results for sampling systems and component measurements are presented for four representative processes: Hot He degas of wafers, high-density (fluorocarbon) plasma etch, and monitoring atomic layer deposition and various CVD processes.

#### 3:00pm VT-MoA4 A Novel Electrostatic Ion Trap Mass Spectrometer, *A.V. Ermakov, B.J. Hinch*, Rutgers University

We have developed, built and tested a novel mass spectrometer which uses purely electrostatic fields for confinement of in-situ ionized residual gases within a linear trap. An anharmonic trapping potential well focuses the ion trajectories of all ion masses, and of a wide range of ion energies, such that their lifetimes are long enough to allow for systematic sequential ejection of the ion mass/charge ratios. Ions are generated within the ion trap by electron impact of residual gases. Mass selective ejection is achieved through a novel autoresonant pumping process. The mass spectrometer has an unlimited mass range, is capable of achieving high sensitivity at high and ultrahigh vacuum levels, and has demonstrated much faster scan rates than the widely used (larger size and) more complicated quadrupole or magnetic sector mass spectrometers. In addition, the new mass spectrometer is very compact (less than 2' long), and requires very low power (in the mW range, excluding ionizer) as it uses only static bias potentials and a very small RF voltage (in the 100mV range). The principles of autoresonant ejection of ions are presented, along with a detailed analysis of the design and operational parameters affecting mass spectral resolution, detection limit, and dynamic range.

#### 3:40pm VT-MoA6 A Practical Electrostatic Ion-Trap Sensor, *G.A. Brucker, J. Rathbone, S. Blouch, M. Schott, K. Van Antwerp*, Brooks Automation, Inc.

We have expanded upon the work of A.V. Ermakov and B.J. Hinch from Rutgers University to further develop a novel electrostatic ion trap sensor that is based upon the principles of autoresonant ion-ejection and detection for use in low mass range mass spectrometry applications. The improved sensor is approximately 15 cm in length, with a 2.5 cm ion-trap component capable of scanning a mass range of 1-300amu with a mass resolving power of  $>130x$ . A symmetric RF pumping scheme combined with a custom RF frequency sweep profile provide simplified electrical design along with optimal ion ejection efficiency across the entire mass range. A novel dual-filament, off-axis ionization scheme provides both pressure range and detection limit enhancements. The entire structure that includes the ionizer, mass analyzer and detector were integrated into a pre-existing Stabil-Ion® gauge total pressure ionization sensor envelope. The improved sensor retained many of the benefits of the original concept sensor such as a rapid sensor scanning speed approximately 70ms for 1-100amu scans and a mW RF ion-trap drive requirement that allows for a remote gauge cable connection to the controller electronics. The simple structure and intelligent electronics were implemented to allow for self-calibration of partial pressure measurements and automated recalibration of the electron multiplier detector. A novel dual-filament ionization source design was incorporated into the ion trap to allow fast and easy field replacement of filaments.

#### 4:00pm VT-MoA7 Total Pressure Measurement Integrated into a Ratiometric Partial Pressure Electrostatic Ion Trap Sensor, *P.C. Arnold, G.A. Brucker*, Brooks Automation, Inc.

A method of total pressure measurement concomitant with partial pressure analysis in a partial pressure analyzer ion trap will be presented along with representative construction geometries and data of the measurements. This combination of total pressure and partial pressures into a single sensor allows a more complete evaluation of the gas content of a vacuum chamber as well as its changing composition and absolute pressure. This method includes one ionization gauge structure which measures the pump-down progress to evaluate correct time of trap turn-on. Another ionization gauge structure is included that provides total pressure at high vacuum intermittently with the partial pressure scans. Ion traps are ratiometric devices that have an upper limit in the ion density that can be stored in their ionization trapping volumes. This limits the total number of ions measurable so that as pressure increases, no further absolute increase is observed in the sum of the amplitudes of individual gas species peaks, even though the differentiation in the various relative mass peaks continues to be readily seen.

#### 4:20pm VT-MoA8 Instrument Design for an Integrated Total and Partial Pressure High Speed Vacuum Quality Monitor Sensor, *M. Schott, J. Rathbone, P. Sandt, K. Van Antwerp*, Brooks Automation Inc.

Design details and performance test results of an integrated total and partial pressure high speed vacuum quality monitor instrument will be presented. A single-sensor, high-speed total pressure and partial pressure instrument has been developed with a 1-100amu, 100ms update rate. The complex sensor is comprised of a hot-filament ion source, electrostatic ion-trap mass separator, Shulz-Phelps based total pressure detector, and an electron

multiplier ion detector that is shared for both UHV total pressure and ratiometric partial pressure measurements. A dual board electronics instrument package has been designed to control, drive and process complex sensory data, and output the processed data into a usable form within the cycle time of the measurement update rate. High speed data processing and real time sensor control was achieved by dividing the sensory control and measurement function from the data reduction and host input/output functions. The mass spectrometer interface (MSI) electronics board utilizes a local uC, which directly controls and provides system status on the following sensory functions: multiple ion-trap bias voltages, filament power & emission current, ion-trap mass analyzer RF drive amplitude & frequency and ion current electrometers. The MSI local processor accepts serial high-level commands from the host input/output (HIO) board processor, which predetermines the sensory "scan" control parameters. The HIO board performs data reduction and user input/output. HIO data reduction algorithms process the raw sensory data into usable total and partial pressure [amu, amplitude] matrix data. All resulting in hard-coded and/or custom user outputs, which may produce a number of real-time and non-real-time datum.

4:40pm **VT-MoA9 NIST Traceable Vacuum Standard Based upon MEMS Resonant Silicon Gauge Technology**, *J. Hendricks, T. Gooding, D.A. Olson*, National Institute of Standards and Technology

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. Over the past decade, NIST has calibrated these gauges and has found them to be very stable, rugged, and ideally suited as core technology for a high-stability precision pressure standard that can be calibrated against the NIST primary pressure standards [1].

The RSGs use two single-crystal silicon resonators encapsulated in a vacuum microcavity. Changes in pressure on the diaphragm are determined by measuring strain-induced changes in the two resonant frequencies [2]. Since each resonant element is encapsulated in a vacuum, the most critical part of the sensor is never in direct contact with the calibration gas which makes the pressure sensor gas species independent. The RSG sensors are commercially available and NIST has performance data dating back over 9 years on one sensor that has been calibrated 18 times, and has a demonstrated average calibration shift of only 0.008% [3].

NIST has developed and built a Resonant Silicon Gauge Transfer Standard Package (RSG-TSP) with a range of 100 Pa to 130 kPa. NIST scientists recently completed a long-term stability study of this transfer standard, demonstrating that the uncertainty due to stability is only a few ppm at 130 kPa, increasing to 0.01 % at 100 Pa ( $k=1$ ). This standard is expected to find applications in national "round robin" and international key comparisons of pressure standards, and is ideally suited for use as a "high end" precision pressure standard for secondary calibration laboratories.

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

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

[3] NIST internal calibration report NC212.

5:00pm **VT-MoA10 A New Approach to Gas Flow Calibration**, *P.D. Levine*, Zero K Designs

Gas flow calibration is typically accomplished by measuring the rate at which volume is displaced by gas flowing at constant pressure. The measurement relies upon the accurate determination of the first order change in position of the object displacing the gas. Through active control of gas pressure and displacement, higher order effects are minimized to enhance measurement precision. These systems are mechanical in nature and require substantial sophistication in their manufacture and utilization to achieve primary standards levels of uncertainty. An alternative method which generates volumetric flow-rates by non-mechanical means is described. The method takes advantage of a natural physical process inherently linear to first order within practical measurement intervals.

By allowing gas to leak from a fixed volume through a throttling valve, leak-down can be controlled such that first order effects dominate for measurement times sufficient for high precision data to be obtained. The volumetric flow-rate is directly proportional to the rate of pressure drop within the fixed volume during the measurement interval. Thus accurate measurements of the fixed volume and the rate of pressure drop can yield high precision results. This method takes full advantage of the resolution and sensitivity offered by state of the art Capacitance Diaphragm Gages (CDG) as used in national standards laboratories. As such, it should be possible to achieve uncertainty levels approaching those of primary references. Indeed, previous work has proven this method as effective as displacement based standards for orifice flow calibration systems[i]. The basic concept lends

itself to many realizations making it capable of covering a wide range of flow rates.

One such realization, which can be readily assembled from off the shelf components and some custom fabrication of flow manifolds, is described. It would be capable of providing calibration to 1000 SCCM (Standard Cubic Centimeters per Minute) with a minimum estimated  $k=2$  uncertainty of 0.60%.

The Precision Gas Flow Calibrator described here represents a new and simplified approach to gas flow calibration. The achievable measurement precision offers the potential of a stable and reproducible reference with great utility for the calibration of a wide variety of gas flow measuring devices.

iP.D. Levine, J.R. Sweda, "A Precision Gas Flowmeter For Vacuum Calibration", *J. Vac. Sci. and Technol. A* 15(3) May/June 1997; pp 747-752.

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