

# Monday Afternoon, October 18, 2010

## Vacuum Technology

Room: Laguna - Session VT+MS-MoA

## Gas Analysis in Vacuum and Process Applications

Moderator: S. Thornberg, Sandia National Laboratories

2:00pm VT+MS-MoA1 **Miniature Mass Spectrometers and Ambient Analysis by Mass Spectrometry**, *R.G. Cooks, R.J. Noll, Z. Ouyang*, Purdue University **INVITED**

2:40pm VT+MS-MoA3 **Accurate Determination of Molar Quantity for Gas in a Vacuum Chamber with Extreme Temperature Variations**, *H.C. Peebles, M.S. Benner*, Sandia National Laboratories, *T.K. Mehrhoff*, Independent Contractor to Sandia National Laboratories

Mass spectroscopy is routinely used to quantify gases in analytical measurements and process diagnostics. In many of these applications, gases enter the ion source of the mass spectrometer through a molecular leak. Sections of the gas manifold on the high pressure side of the leak may contain extreme temperature gradients such as a gas source operating at a very high temperature or a cryogenic trap at low temperature with the remaining manifold components near room temperature. Calibration of the mass spectrometer response to the molar quantity of gas present in the manifold must take into account the thermal gradient over pressures that can span the range from viscous flow to the molecular flow regime where thermal transpiration dominates. This paper will present a method for calibrating the molar quantity of gas present in a manifold with a large but constant temperature gradient using a pressure gauge and molar calibration source (MCS) attached to a room temperature section of the manifold. The MCS is a calibrated gas volume and integrated pressure gauge maintained at constant temperature. Molar quantities of gas released from the MCS into the manifold are related to the response of the pressure gauge using empirical n/P functions. These functions accurately relate the manifold pressure to the molar quantity of gas in the manifold as long as the temperature distribution across the manifold remains stable. An example will be presented showing the application and stability of n/P functions used in thermal desorption measurements of hydrogen isotope concentrations in thin metal films. The construction and critical performance characteristics of the MCS will also be described.

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3:00pm VT+MS-MoA4 **Hydrocarbon Measurements at ppb Level at 10Pa Absolute Pressure**, *R. Versluis, M.F. Dekker*, TNO Science and Industry, Netherlands

In Extreme UltraViolet lithography (EUVL), 13.5 nm light is used for imaging the reticle pattern onto the wafer. Since EUV light is absorbed by all materials (including gases) a reflective optical system is used in EUV wafer steppers. Wafer resist outgassing during exposure is one of the most important contributors to mirror contamination. Just one or a few monolayers of Carbon on the multi-layer reflective optics of the system leads to an unacceptable reflectivity loss. Considering the fact that a large fraction of the EUV induced wafer resist outgassing constitutes of hydrocarbons, suppression and mitigation of these molecules is absolutely necessary.

Different methods exist to suppress hydrocarbons and other contamination from wafer, or 'dirty' components. In order to qualify such methods, very sensitive hydrocarbon measurements need to be done at relatively high pressures.

We built a test rig capable to detect hydrocarbons at ppb level. The hydrocarbon levels that need be measured are at the order of  $1E-9$  Pa, while the absolute pressure is at the order of 10 Pa. The test rig therefore needs to be extremely clean during the measurements. The test rig includes six large Turbo Molecular Pumps to keep the test rig at an absolute pressure of 10 Pa, while the component under test is continuously being flushed. The electro-polished setup (about 4 meters long and one meter high) can be completely baked at 150°C to reach the extreme clean conditions needed to measure the low concentrations of hydrocarbons. Measurements are done with a very sensitive residual gas analyser (RGA) which can be baked to 200°C. The RGA has been optimized in such a way that the sensitivity for heavy gases is increased with respect to the sensitivity for lighter gases (patent appointed). Test gases (hydrocarbons, Ar, N<sub>2</sub> can be injected at different

locations in the test rig. This way a contamination source in the wafer stage as well as wafer resist outgassing can be simulated.

With the test rig we can also do a thermal qualification of the component under test, to determine heat loads to the test component and heat load distributions.

The presentation will focus on design and engineering aspects of the test rig, the qualification of the test rig and the qualification of the component under test.

3:40pm VT+MS-MoA6 **Ratiometric and Absolute Partial Pressure Measurements with Low Mass Range Mass Spectrometers**, *G. Brucker, J. Rathbone, K. Van Antwerp, M.N. Schott*, Brooks Automation, Inc.

Low mass range mass spectrometers are routinely used to obtain partial pressure information in high vacuum and ultrahigh vacuum systems. Absolute and ratiometric partial pressure measurements are both applied to monitor and control vacuum processes and experiments. Mass spectrometers provide indirect partial pressure measurements, and require advanced data interpretation and analysis procedures in order to generate accurate partial pressure measurements from their raw spectral output. This presentation describes some of the modern methodologies used by commercial equipment manufacturers to derive accurate absolute and ratiometric partial pressure information using both quadrupole mass spectrometers and a new generation of electrostatic ion trap mass spectrometers. The advantages of combining accurate total pressure readings with native ratiometric partial pressure information from electrostatic ion traps is also described and compared to standard partial pressure measurement methodologies. Fast process control, in the millisecond timescale, based on partial and total pressure measurements is also described.

4:00pm VT+MS-MoA7 **Accurately Modeling the Natural Frequencies of Ions Ejected from an Anharmonic Resonant Ion Trap**, *M.N. Schott*, Brooks Automation, Inc.

A mathematical model is demonstrated that accurately predicts the natural frequencies of ions ejected from an Anharmonic Resonant Ion Trap (ion trap). The model is based upon a force balance equation of motion, which is comprised of a forcing function, a mass, a damper and a spring. The forcing function is a low amplitude RF signal, swept from high frequencies to low frequencies over a given period, which locks-up, bunches and then ejects ions with a common mass-charge ratio using the principal of autoresonance. The ionized gas particles provide the mass portion of the equation, where specific autoresonant mass selection is dependent upon the selected ion's mass-charge ratio. The equation damping, or ion trap losses, are primarily dependent upon ion trap pressure. The spring in this model is provided by the trap's static anharmonic voltage gradient, which is a function of ion trap geometry and the associated relative-voltage-potentials distributed across the ion trap's geometric structures. The ejected ions natural frequencies are then proportional to the square root of the anharmonic voltage gradient, the pressure dependent damping and the autoresonant selected mass-charge ratio. Experimental results are presented that vary the independent anharmonic voltage gradient, pressure, and ionized gases versus dependent ejected ion natural frequencies on the ion trap compared with the model's predicted natural frequencies.

4:20pm VT+MS-MoA8 **Solving the Low Mass Range Mass Spectrometer Limitations (Zero Blast) using Electrostatic Ion Traps**, *P.C. Arnold, G. Brucker, J. Rathbone*, Brooks Automation, Inc.

The phenomena of typically unavailable resolution of low mass spectroscopic peaks (zero blast) will be presented along with a solution that allows clear resolution of those low mass peaks, for example, masses 1 to 4. A new mass spectrometer design composed of an electrostatic ion trap using anharmonic resonant trapping potentials will be shown to resolve this issue. The primary causes of poor low mass resolution will be presented. The background of the physical electronics of the problem will be discussed. The new mass spectrometer design will be described with respect to its effect on zero blast. A test program to demonstrate the solution and show results at scans of low mass will be presented.

4:40pm VT+MS-MoA9 **Ion Residence Times for Electron-Impact Ion Sources of Mass Spectrometers**, *R.E. Ellefson*, REVac Consulting, *M.F. Vollero*, INFICON, Inc.

Electron impact ionization is a common method of ion production for mass spectrometers. The mass spectrum produced is affected by the electron energy and ion residence time in the ionization region. The residence time is the time between the initial direct ionization event and the extraction and focus of the ion(s) from the ionization region into the mass analyzer. During

the residence time, additional ionization, fragmentation and ion-molecule reactions can occur. In closed ion sources or other high-pressure ion sources, ion-molecule reactions can be significant; the ions extracted and analyzed can exhibit a bias in inferred gas composition due to the addition or depletion of the species-related ions of interest by the ion-molecule reactions. The presence of a potential well created by the ionizing electron beam and the ion extraction potentials dictate the residence time for ions in the ion source. Models for the depth of the potential well as a function of electron emission current and ion extraction potentials are given and estimated ion residence times are calculated. Evidence for change in residence time with emission current is provided by monitoring mass 80- $\text{Ar}_2^+$  abundance relative to mass 40- $\text{Ar}^+$  at a fixed ion source pressure for different emission currents. Other ion-molecule reactions (e.g.  $\text{N}_2^{+*} + \text{N}_2$  gives  $\text{N}_3^+ + \text{N}$ ) and charge-exchange reactions (e.g.  $\text{He}^+ + \text{Ar}$  yields  $\text{He} + \text{Ar}^+$ ) are presented together with pressure dependence and magnitude of the reactions. Recommendations are given for ion source operation to minimize biases in compositional analysis of gas mixtures.

5:00pm **VT+MS-MoA10 Performance Characteristics of a New Wide Range, Fast Settling Electrometer Design for a Residual Gas Analysis Mass Spectrometer**, S. Billington, MKS Spectra Products UK, J. Blessing, MKS Instruments, R. Fletcher, P. Shaw, MKS Spectra Products UK

The use of faraday or electron multiplier detectors in mass spectrometry has always presented the electronics engineer with the challenge of having to choose some compromises in a design for the electrometer. The maximum measurable signal usually determines the value for the feedback resistor that is required. However, with a conventional electrometer where a high impedance input Operational Amplifier is used in an inverting configuration with a feedback resistor, the noise is dominated by the feed-back resistor value. A typical quadrupole mass spectrometer design is capable of generating partial pressure ion currents from a scan of masses which encompasses the full range of the detector output. In order to utilise this full range, more than one feedback resistor is used with switching between gain ranges or a logarithmic amplifier is used. Intrinsicly, the logarithmic amplifier requires significantly longer settling times for lower signal levels which tends to make it impractical for a mass spectrometer where two signals at the extremes of the dynamic range of measurement can be only a few milliseconds apart. Traditionally a gain switching electrometer has been the choice of RGA designers but this has resulted in the compromise of having to either choose which gain range to use for a particular scan of masses or wait for several tens of milliseconds each time the range is switched during the scan.

This work will describe a new electrometer design which allows the use of two gain ranges in a scan of masses with settling times of less than 20ms per measurement point. Data will be presented to show the effectiveness of the design for speed of measurement and the wide dynamic range available. RGA data will also be shown of common applications that traditionally would have required a compromised speed of acquisition or reduced dynamic range. Data will also be shown on the improved accuracy offered for fast transient peak measurements with the faster data acquisition rates of the new RGA design.

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