# Thursday Afternoon, November 5, 1998

# Partial Pressure Measurements and Process Control Topical Conference

### Room 317 - Session PC-ThA

#### **RGA Characteristics and Calibration**

Moderator: S.A. Tison, Millipore Corporation

#### 2:00pm PC-ThA1 Semiconductor Applications of a Quadrupole Mass Spectrometer, R.K. Waits, MKS Instruments INVITED

Commercial quadrupole mass spectrometers (QMSs) became available in the late 1960s and have been popular in R&D labs, but until recently have found limited use in semiconductor manufacturing. To sample at pressures above 10@super -5@ Torr with ppm sensitivity or better (relative to the total process pressure), differential pumping is usually required. The newlyavailable, small, high-pressure QMS sensors can operate as high as 10 to 20 mTorr without differential pumping, but provide somewhat lower mass resolution and partial pressure sensitivity than a standard QMS. Applications in the semiconductor fab include equipment monitoring, process monitoring and effluent analysis. Equipment monitoring can include gualification after preventative maintenance, rate-of-rise tests, and leak identification and detection. Usually the burning question is: Why won't the vacuum chamber pump down? Other uses that are not usually considered include the qualification of replaceable parts: sputter cathodes, electrodes, lamps, shields, etc. The trend to smaller features and thinner layers on larger, more expensive wafers requires better in-situ monitoring of fabrication processes. In process monitoring, the key question is: Is this process running normally? A manufacturing monitor can be useful simply by providing a comparison between a well-behaved high-yield process and a marginal or failing process. Experience mixed with a little process expertise can link the symptoms, as shown by QMS spectra, with the root cause of the disease, result in a prompt cure, and lead to continuous process improvement. Examples will be given for physical vapor deposition (sputtering) processes, chemical vapor deposition and plasma etching. The effluent from chemical vapor deposition and plasma etch processes can be analyzed to measure the efficiency of process gas utilization or to monitor the efficacy of abatement methods used for the removal of global warming gases.

#### 2:40pm PC-ThA3 In Situ Monitoring of Semiconductor Reactive Gas Processes using Partial Pressure Analyzers, *L.C. Frees*, Leybold Inficon, Inc. INVITED

As semiconductor fabrication is pushed towards narrower linewidths utilizing new materials, processes such as chemical vapor deposition (CVD) and etch increasingly employ reactive gases. These gases, along with high temperatures and/or plasmas, and process pressures ranging over six orders of magnitude (1E-1 to 1E+5 Pa) present considerable challenges to the partial pressure analyzers (PPAs) and systems used to monitor them. Techniques used in the design and construction of the sample inlet system, the differential pumping system and the PPA itself which result in a viable in situ process monitor will be discussed. Emphasis will be given to the ion source itself. Choices concerning the place on the process tool to connect the PPA, and their effects on the data obtained, will also be covered. Applications examples will include CVD of the metals Cu. Ti (and TiN), and W and Al. Also included will be the CVD of dielectrics such as silicon nitride and phosphosilicate glass. Sampling methods for monitor etch processes for both metals and dielectrics will be presented, with a focus on the lifetime of the ion source.

#### 3:20pm PC-ThA5 Emission Free Measurement of Residual Gas in XHV Using Ionization by Trapped Electrons in Magnetic Field, A. Yamamoto, S. Kato, KEK, Japan

One problem associated with partial pressure measurement in an extremely high vacuum (XHV) region is outgassing from an ion source of a residual gas analyzer (RGA) itself. In order to reduce the outgassing, an improvement of its structural materials of the ion source was reported previously.@footnote 1@ However there still remains a problem of thermal outgassing from the ion source as far as a hot filament is used. Therefore, it is required for suppressing thermal outgassing to limit a time of electron emission from a filament. In this work we used a hot filament for a limited time in the beginning of the measurement. Adopting an axial magnetic field to a cylindrical anode to make a flight time of emitted electrons long, we could keep electrons trapped inside. These trapped electrons allowed us to ionize the residual gas without the thermal outgassing. But dwindling of the number of electrons due to the electron -

gas collisions leads to decreasing of ion currents. We measured a dependence of the decay of ion currents for He, Ne, N2 and Xe on a gas pressure in a range of 10@super -7@ to 10@super -8@ Pa. And we also compared the decay of ion currents for the different gas species in the same pressure range. We verified that the decay time decreased with an increase of the pressures or the molecular diameters. @FootnoteText@ @footnote 1@S.Watanabe, M.Aono, S.Kato, J. Vac. Sci. Technol. A 14, 3261 (1996).

#### 3:40pm PC-ThA6 Residual Gas Analyzer Ion Current Measurement, Calibration and Partial Pressure Detection Limits, *R.E. Ellefson*, *A.J. Kubis*, *L.C. Frees*, Leybold Inficon, Inc.

Ion detection in a residual gas analyzer (RGA) is by faraday detector with electrometer and/or a secondary electron multiplier detector that use the same or separate electrometer. The minimum detectable partial pressure (MDPP) measured by these detectors is a ratio of noise(A) of the detection system to the sensitivity(A/Torr) of the RGA for each detector type. Critical to the statement of MDPP is the inclusion of the integration (dwell) time interval used to determine the noise value. Usually the MDPP reported is the longest integration time period of the RGA which produces the lowest number. However, the user normally uses integration times of the order of 0.25 s or less to rapidly get data for timely observation of the process. In this paper we present a model for predicting MDPP as a function of integration times from 8 ms to 4 s based on detector noise and ion statistics. Separately we present ion current measurements of the @super 36@Ar and @super 38@Ar minor isotopes of argon as a function of pressure to demonstrate practical detection limits of a RGA as a function of integration time. Additionally, we present data from the systematic dilution of standard gas mixtures and from a fixed composition flow standard that validate the low ppm detection limits for impurities in Ar.

#### 4:00pm PC-ThA7 Practical Quadrupole Theory: RGA Characteristics, R.E. Pedder, ABB Extrel INVITED

Residual Gas Analyzers (RGA's) are commonly used to monitor the partial pressures of contaminants, process gases and reaction gases in various vacuum processes. Quadrupole mass filters can be used as RGA's through the application of RF and DC voltages in such a way as to make ions of a single mass or narrow range of masses to transmit through the quadrupole to the detector. The physics that describes the trajectories of these ions through this electrodynamic field is well studied. The performance characteristics that can be inferred from such trajectories have been predicted through both analytical and numerical methods. Unfortunately, the mathematics involved, while straightforward, is often beyond the comfort level of the practical experimentalist. This presentation will include a broad review of practical quadrupole theory, utilizing graphical means to illustrate the indicators to quadrupole performance, and avoiding all but the most straightforward equations. The goal of this presentation is to provide a more intuitive understanding of quadrupole operation, with emphasis on practical issues. Key performance figures of merit will be identified along with a practical analysis of the theoretical indicators to performance (e.g. transmission/sensitivity is proportional to the square of the rod diameter, resolution and abundance sensitivity increase with increasing RF frequency). Performance characteristics of a wide spectrum of analyzers will be compared. The performance compromises that are inherent in the optimization of quadrupole analyzer characteristics for a given application will be reviewed.

# 4:40pm PC-ThA9 Residual Gas Analyzer Performance Characteristics, C.R. Tilford, National Institute of Standards and Technology; T. Gougousi, University of Maryland

Reliable process monitoring and control require reliable instrumentation. Residual gas analyzers (RGAs) are promising candidates for these applications, but only if they are properly adjusted and used. The National Institute of Standards and Technology's (NIST) earlier work on the performance characterization and calibration of conventional, or opensource RGAs, is being extended in collaboration with the University of Maryland. This new work includes the characterization of closed-source RGAs, the development of in situ RGA calibration techniques for use in a CVD tungsten deposition tool, and the application of the calibrated RGAs in the monitoring and control of the tungsten deposition process. This talk describes fundamental characteristics of RGAs that limit their performance, and techniques to detect and minimize these undesirable characteristics. Particular attention is paid to operating conditions that cause the sensitivity for one gas to depend on the pressures of other gases.

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5:00pm PC-ThA10 Calibration of Gas-Analytic Mass Spectrometers for Gases and Vapors, *R. Dobrozemsky*, *G.W. Schwarzinger*, Vienna University of Technology, Austria

The demand to quantify pressure-, density-, and flow-rate-readings is steadily growing, e.g. for quality control. By many reasons, simple and reliable in-situ calibration methods for pressure-reading instruments (e.g. BA-gauges) and partial pressure analyzers (e.g. quadrupole mass spectrometers) are required. In this contribution, the potential of in-situ methods for calibration of vacuum instruments is discussed, with special attention on admitting gas bursts, defined by expansion of known quantities of gases and vapors. Ten years ago, a "gas-burst calibration" procedure for non-reactive gases has been introduced at Seibersdorf.@footnote 1@ Recent demands in geological research and space technology led to new calibration procedures for water- and oilvapors (thermal decomposition method - TDM and crack-product calibration - CPC, respectively). By these methods, in-situ calibrations can be done with an accuracy of 1 to 3% for non-reactive gases (e.g. H@sub 2@, N@sub 2@, CO, CO@sub 2@, CH@sub 4@, He, Ar, etc.), of about 10% for water vapor and of about 20 to 40% for oil vapors. Moreover, calibrations can be repeated several times a day, if necessary (e.g. under harsh conditions), and ca be performed in a wide pressure range down to uhv. @FootnoteText@ @footnote 1@ R. Dobrozemsky, Vacuum 41, 2109 (1990)

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