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 newly-available, 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 qualification 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 open-source 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.

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 oil-vapors (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)

Partial Pressure Measurements and Process Control Topical Conference
Room 317 - Session PC-FrM

Process Monitoring and Control

Moderator: M.L. Passow, IBM Corporation

8:20am **PC-FrM1 Challenges for Real-Time Control in Reactive Semiconductor Manufacturing Process Environments**, ***G.W. Rubloff***, University of Maryland **INVITED**

The potential of mass spectrometry and other chemical sensing approaches to determine dynamic chemical constituency through a reactive semiconductor process cycle holds substantial promise for process control and attendant manufacturing benefits. Applications to mainline VLSI processes have already demonstrated value in supplying metrology information (rates, deposition thickness, etch endpointing) and in revealing process mechanisms and equipment subtleties, information which significantly complements the existing raison-d'etre for RGA's in manufacturing, namely, equipment qualification and debugging. However, the benefits of advanced process control will require other elements. Use of sensor signals for metrology depends on having good sensor models, which in turn often means good models for the sensor IN the tool. Real-time or run-to-run control necessitates either good models or robust algorithms which are less sensitive to model or data error. Fault management - i.e., fault classification and prognosis as well as detection - requires multi-sensor integration including equipment state signals for decision support to minimize down-time, along with dynamic models for interpreting system behavior. This talk will provide examples for mass spec based metrology and process insight, along with an outline of how sensor information might lead to beneficial control and fault management responses.

9:00am **PC-FrM3 Reaction Analysis and Rate Metrology of Selective Area Silicon PECVD using In-Situ Real-time Mass Spectroscopic Sensing and Mass Balance Modeling**, ***A.I. Chowdhury***, *T.M. Klein, G.N. Parsons,* North Carolina State University

Thin film processes in microelectronics fabrication often have intermediate steps that need to be quantified for optimization purposes. These intermediate steps, including concurrent etching and deposition cannot be quantified using only final state analysis. Additional real-time process state data is required. Mass spectroscopy is a useful process state sensor for SiH@sub 4@ processes because it is particularly sensitive to changes in silane concentrations in the sampled gas. We use real-time in-situ mass spectroscopy and mass balance modeling to quantify deposition and etching reaction rates in a cyclic deposition/etch process that leads to selective area microcrystalline silicon PECVD. The procedure involves repeated cycles of a SiH@sub 4@/He/H@sub 2@ plasma followed by a He/H@sub 2@ plasma. In order to monitor reactant concentrations in real time, process trace data were collected at 30 amu corresponding to SiH@sub 2@@super +@, the principal SiH@sub 4@ related signal generated in the ionization region of the mass spectrometer. We have developed a mass balance model that can be used in conjunction with real-time sensor data, such as mass spectroscopy, to quantify deposition and etching rates in selective deposition. During SiH@sub 4@/He/H@sub 2@ flow, when the plasma is initiated, we observe a decrease in the silane signal that is correlated to film deposition. During the He/H@sub 2@ plasma the silane signal is larger when the plasma is on, and the change is a quantitative indicator of silane produced by etching. OES data does not show comparable sensitivity to silane concentration changes for our process. The transition from selective to non-selective conditions can be detected in real-time. A sharp change in slope of the silane signal is observed during etching, which is consistent with complete removal of stray nuclei from the non-receptive surfaces. Fitting the real-time mass spectroscopic data to the mass balance model we calculated the etch rate to be ~2.1 nm/min on receptive surfaces such as c-Si and ~4.5 nm/min on non receptive surfaces such as SiO@sub 2@. The values are consistent with other results. This demonstrates rate sensitivity for intermediate process steps. The mass balance analysis also reveals that these rates are obtainable only if real-time process sensor data is available.

9:20am **PC-FrM4 Sensor Integration on a W-CVD Cluster Tool for Real-Time Process Monitoring and Control**, ***J.N. Kidder, Jr.***, *Y. Xu, N. Gupta, T. Gougousi, G.W. Rubloff,* University of Maryland

Research in in-situ chemical process sensing and sensor integration is motivated by the potential value of real-time and in-line sensing for metrology, control, and optimization. In this work, the chemical composition of a W-CVD process flow downstream of the reactor was analyzed using a differentially-pumped closed ion source mass spectrometry system. LabView software and data acquisition hardware were employed to integrate equipment state signals (total pressure, valve status, temperature, etc.) with mass spectrometer measurements so that time-synchronized system behavior was obtained and the relation between the equipment and process variables was established. The sensor integration allowed us to monitor the reaction process via the chemical composition of the gas-phase reaction products while capturing the dynamics of the reactant delivery and pump behavior through the process cycle. Generation of product species from the W nucleation and growth stages as well as other time-dependent variations in the downstream process flow composition were detected with rapid response time (~2 s), which provided insight to the reaction dynamics at critical stages in the process. In addition, this sensor fusion facilitates identification of subtleties like flow rate fluctuation and run to run effects which are important for process design and fault management.

10:00am **PC-FrM6 In situ Measurement of Moisture Contamination in Reactive Process Atmospheres**, ***J.J.F. McAndrew***, *R.S. Inman, D. Znamensky,* Air Liquide*; J.-M. Girard,* Air Liquide, France*; G. Goltz,* France Telecom, France*; J.-M. Flan,* SGS-Thomson, France

In situ measurement of molecular contamination in semiconductor processing is important for (i) early detection of contamination events to avoid accidental misprocessing (ii) real-time control of purging procedures to improve tool utilization, and (iii) understanding of actual contamination levels in process atmospheres. In the present work the primary goal is to simplify chamber qualification and process more wafers between maintenance. Semiconductor processing applications use reactive atmospheres with which in situ monitoring equipment must be compatible. Achieving real benefits in a manufacturing environment requires a sensor which is user-friendly and reliable, even under demanding conditions. Absence of drift and reliability of calibration are also important. In order to simplify implementation, it is desirable to monitor as few species as necessary to obtain the desired information. Water vapor may be used as a general diagnostic of ambient contamination, because it is present in relatively high concentration in air and is often the most difficult species to eliminate from the process atmosphere. The implementation of Tunable Diode Laser Absorption Spectroscopy (TDLAS) for in situ monitoring of water vapor in a Rapid Thermal Processor (RTP) has been described previously.@footnote 1@ That work did not address compatibility with reactive atmospheres, as the RTP uses only nitrogen as process gas. Here, we will describe the implementation of TDLAS in aggressive environments, including application to silicon nitride CVD and other processes. Implementation of the TDLAS system in a CVD process poses significant challenges, as deposition of reaction products on the sensor optics must be carefully limited. In silicon nitride CVD, appropriate heating of the optics has been found to be critical. Water vapor levels measured during processing and their impact on process parameters will also be described, as will the use of real-time contamination measurement to improve tool-utilization by simplifying chamber qualification. @FootnoteText@ @footnote 1@J.J.F. McAndrew R. S. Inman, A. Haider and J. Brookshire, 44th International Symposium of the AVS (1997) Abstract Number: 1159: Program Number: MS+VT-ThA7 (submitted to JVST A)

10:20am **PC-FrM7 RGA Process Monitoring in Semiconductor Manufacturing**, ***J.M. Baker***, IBM T.J. Watson Research Center **INVITED**

Although RGA's have been a mainstay in vacuum technology for decades, the semiconductor industry has been slow in realizing their potential for tasks other than leak checking and vacuum troubleshooting. This is in large part because the pace and culture of the manufacturing environment provides neither the time nor the skill to deal with the combined complexities of a process tool plus an RGA system. To succeed in this environment, it is necessary to extend the basic sensor technology and build the infrastructure so that the sensor is tightly integrated with the process tool and can adapt to the process and product types. I will show how we have addressed some of these obstacles in IBM fabs through a combination of distributed hardware and software. By incorporating tool signals with the data collection software, we have been able to detect and

stop contaminated wafers at a degas station in the presence of interfering pressure bursts. Through links to factory/tool control software, we have been able to obtain recipe information and adjust our monitoring methods to different process chemistries. With this capability, we have been able to collect extensive data and observe a variety of interesting and often unexpected behavior, examples of which will be presented. In concluding, I will discuss some of the implications of our experience for utilizing versatile sensors such as RGA's as part of an advanced fault detection or process control system and the importance of having the information infrastructure necessary to closely integrate with the tool, the process, and ultimately the product.

11:00am **PC-FrM9 Process Monitoring of Chemical Vapor Deposition Systems by In-situ Gas Analysis**, ***B. Lu***, *E. Baker,* Novellus Systems, Inc. **INVITED**

Chemical vapor deposition (CVD) is a key technology for both interconnection and front-end wafer processing in advanced IC fabrication. CVD systems present a challenge for process state monitoring due to the inherent complexity of the process chemistry involved. When combined with other process knowledge, in-situ gas analysis using mass spectrometry (typically referred to as RGA) can provide real-time information representing the process chemical composition, process sequence, and equipment status. Consequently, such process monitoring tools can aid significantly in the continuous improvement and new product development of semiconductor processes and process equipment. Several applications will be presented to illustrate the use of a properly configured mass spectrometer on CVD process systems for (1) rapid learning of process chemistry and process state, (2) verification of system design improvement, and (3) optimization of loadlock operation to eliminate residual air contamination. We will also discuss some of the current obstacles that must be overcome in the use of mass spectrometer as a "full-time" process state sensor.

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