

# Monday Morning, October 18, 2010

## Vacuum Technology

Room: Laguna - Session VT+MN-MoM

## MEMS Sensors, Vacuum Gauges, Measurements and Pumps

**Moderator:** J. Fedchak, National Institute of Standards and Technology

8:20am **VT+MN-MoM1 Practical Issues and Applications for Vacuum and Hermetic Microsystems Packaging**, *L. Fang, D. Chu, K. Ewsuk*, Sandia National Laboratories **INVITED**

Microsystems packaging involves physically placing and electrically interconnecting a microelectronic device in a package that protects it from and interfaces it with the outside world. When the device requires a hermetic or controlled microenvironment, it is typically sealed within a cavity in the package. Sealing involves placing and attaching a lid, typically by welding, brazing, or soldering. Materials selection (e.g., the epoxy die attach), and process control (e.g., the epoxy curing temperature and time) are critical for reproducible and reliable microsystems packaging. This paper will review some hermetic and controlled microenvironment packaging at Sandia Labs, and will discuss materials, processes, and equipment used to package environmentally sensitive microelectronics (e.g., MEMS and sensors).

9:00am **VT+MN-MoM3 New NIST Comparison Method Calibration Service for Vacuum Gauges Based on MEMS Pressure Sensors**, *J.H. Hendricks, D.A. Olson*, NIST

A new calibration service based on a secondary pressure transfer standard spanning the pressure range from 0.65 Pa to 130 kPa (5 mTorr to 975 Torr) has been developed at NIST. Until now, vacuum gauges in this range could only be calibrated using the NIST Ultrasonic Interferometer Manometers (UIMs). However, many customers desire direct traceability to NIST but cannot justify the cost of the NIST UIM calibrations. The new service follows a similar model of other calibration services where a lower cost, and less accurate service is offered to customers who do not require the lowest uncertainty possible but still desire direct NIST traceability. The comparison method utilizes a high accuracy transfer standard package that consists of a 133 Pa (1 Torr) Capacitance Diaphragm Gauge (CDG), a 13.3 kPa (10 Torr) CDG and a 130 kPa (975 Torr) MEMS type Resonance Silicon Gauge (RSG) all encased in a temperature controlled enclosure that is periodically calibrated against the NIST 160 kPa UIM and 140 Pa oil UIM primary pressure standards. Due to the superior calibration stability of the MEMS based RSG, the transfer standard package, and ultimately the comparison method vacuum gauge service, provides expanded uncertainties as low as 0.05 % from 1.33 kPa to 130 kPa (10 Torr to 975 Torr) and 0.3 % from 1.33 Pa to 1.33 kPa (0.01 Torr to 10 Torr).

9:20am **VT+MN-MoM4 Pirani for Industrial Processes**, *B. Andreas, R. Enderes, M. Wuest*, INFICON Ltd, Liechtenstein

In modern Pirani heat transfer gauges a filament is usually kept at a constant temperature and the necessary heating power is measured as a function of pressure.

Pirani gauges operated in coating and etching applications suffer from degradation due to process contamination or corrosion. Eventually, the Pirani may fail because the filament is etched away, its resistance and/or its emissivity have changed. Standard procedure for those processes is to use a corrosion resistant filament material adapted to the process in question, e.g. Nickel. Yet the choice of suitable filament material is limited as it needs to be manufactured as very thin coils, be electrically conducting, have a high and well-defined temperature coefficient for the resistance and be chemically inert. For some of the latest manufacturing processes in semiconductor industry none of the available filament materials is sufficiently resistant against corrosion.

We will present here a different approach in that we present first data on a coated Pirani sensor. The coating allows for a much broader field of application as electrical and mechanical requirements of the filament are separated from its chemical properties. Chemical stability is solely due to the coating, all other requirements, unaffected by the coating, can be met using a standard filament material.

9:40am **VT+MN-MoM5 Nitrogen Incorporated Ultrananocrystalline Diamond as a Robust Cold Cathode Material for Miniature Mass Spectrometry Application in Space Exploration**, *X. Wang*, University of Puerto Rico; Argonne National Laboratory, *S. Getty*, NASA Goddard Space Flight Center, *A.V. Sumant, O.H. Auciello*, Argonne National Laboratory, *D. Glavin, P. Mahaffy*, NASA Goddard Space Flight Center

Ultrananocrystalline diamond (UNCD) thin films have been investigated for over a decade for application to electron field emission devices since they offer very low threshold voltage (1-3 V/ $\mu\text{m}$ ) and reasonably stable field emission with time. Due to the small grain size (2-5 nm) and unique atomically abrupt grain boundary structure containing mixed  $sp^2/sp^3$  carbon bonding, it has been postulated that field emission occurs mainly at the grain boundary due to the high field enhancement effect at the grain boundary and stable field emission has been observed independent of surface geometry or film thickness. In addition to low power consumption and potential for miniaturization, robust field emission materials are compelling for applications as long life electron sources for mass spectrometers for space exploration where electron sources are exposed to harsh environments. A miniaturized mass spectrometer under development for *in situ* chemical analysis on the moon and other planetary surfaces requires a robust, long-lived electron source, to generate ions from gaseous sample using electron impact ionization. To this end, we have explored the field emission properties and lifetime testing of nitrogen-incorporated ultrananocrystalline diamond (N-UNCD). The N-UNCD films were synthesized in a microwave plasma chemical vapor deposition system by introducing nitrogen in the Ar/CH<sub>4</sub> gas chemistry. Characterization of the N-UNCD films were carried out by using visible and UV Raman spectroscopy confirming characteristic signature of a good quality N-UNCD film. We will present results revealing that UNCD films with nitrogen incorporation during growth yield stable/high field-induced electron emission in high vacuum for up to 1000 hours.

This work was done with support from the NASA Astrobiology Science and Technology Instrument Development Program, under Grant Number 07-ASTID07-0020, and the NASA Goddard Space Flight Center Internal Research and Development Program. Use of the Center for Nanoscale Materials was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

10:40am **VT+MN-MoM8 The Pumping Synergies of Integrated NEG and SIP Pumps for UHV Applications**, *A. Bonucci, A. Conte, L. Caruso, L. Viale, P. Manini*, SAES Getters S.p.A., Italy

A variety of vacuum systems, such as particle accelerators, synchrotrons, surface science chambers or laboratory equipments, do require the achievement of very high or even extremely high vacuum conditions (UHV-XHV). To this purpose, Ion pumps and Non Evaporable Getter (NEG) technologies are widely applied, since they complement each other effectively. Ion pumps remove ungetterable species like noble gases and methane, while the NEG provides a constant and large pumping speed for all the other gases, in a very compact volume.

So far ion and NEG pumps have been mostly used as separate units mounted in separate part of the vacuum system. In this paper, we investigate how overall pumping performances are influenced by the mounting geometry of the two pumps. In particular we will show that a remarkable synergic effect arises when the two pumps are integrated into one unit having optimized design, known as NEG<sup>+</sup>.

This configuration allows to minimize the detrimental effect given by outgassed species released by the SIP [<sup>1,2</sup>] This increases the real pumping speed of the SIP, generally masked in the UHV range by gas desorption from SIP internal surfaces. This effect is particularly noticeable for ungetterable gases like Argon and Methane.

The resulting pumping speed of the NEG<sup>+</sup> is therefore larger than the sum of the pumping speed of the two separated pumps.

Reducing the degassing effect also increases the overall pumping efficiency at the lower pressure.

In fact, the presence of oxides and nitride compounds onto the cathode surface are known to slow down the diffusion of hydrogen and helium into the cathode [<sup>3</sup>], which is mainly an ion implantation driven process.

In the present paper we discuss some of these effects as well as the synergies arising from the NEG<sup>+</sup> integrated design. A specific focus will be given to argon [<sup>4</sup>] and methane, which are important gases to consider in a variety of application including electron microscopy and electron/ion optics.

[[1]] K.M.Welch, D.J.Pate and J.Todd, "Pumping of helium and hydrogen by sputter-ion pumps. II. Hydrogen pumping", *J.Vac.Sci.Technol.A* 12(3), May/June 1994

[ 2]A Calcatelli et als. "Study of outgassing of sputter-ion pump materials treated with three different cleaning procedures", *Vacuum* vol. 47 n. 6-8, 1996

[ 3]M. Audi and M. De Simon, "The influence of heavier gases in pumping helium and hydrogen in an ion pump", *J. Vac. Sci. Technol. A* 6 (3), May/June 1988

[ 4]D. Andrew, D.R. Sethna and G.F. Weston, "Inert-Gas pumping in a magnetron pump", *proc. 4th AVS*, (1968)

11:00am **VT+MN-MoM9 NEG+: A Novel Route to Compact, High Performance Pumping in UHV-XHV Vacuum Systems**, *P. Manini, A. Bonucci, A. Conte, L. Viale, L. Caruso*, SAES Getters S.p.A., Italy

11:20am **VT+MN-MoM10 Direct Simulation Monte Carlo Modeling of Miniature Vacuum Pumps**, *B.J. Davis, R.W. Hill, P.H. Sorensen, R.J. Kline-Schoder*, Creare Incorporated

NASA and other organizations have pressing needs for miniaturized high vacuum systems. Recent advances in sensor technology at NASA and commercial laboratories have led to the development of highly miniaturized time-of-flight, quadrupole, and ion trap mass spectrometers. However, high vacuum systems of adequate performance continue to be too large, heavy, and power hungry for man-portable mass spectrometers or spectrometers deployed on UAVs, balloons, or interplanetary probes. Terrestrial, man-portable applications impacted by this problem include military and homeland defense systems for detecting hazardous materials as well as portable leak detectors for commercial use.

For over 10 years, Creare has been developing the technologies required to design and build miniature high vacuum pumps. We have designed and built pumps that are as small as a D-cell battery, reach an ultimate pressure of  $10e-7$  torr, have a flow rate in excess of 5 L/s, and spin at 200,000 RPM. As mass spectrometers are reduced in size, the vacuum system requirements can be relaxed. As a consequence, Creare is developing an extremely low-cost and rugged high vacuum system whose performance is optimized for miniature mass spectrometers. The vacuum system is based on an innovative molecular drag pump designed to match the requirements of portable analytical instruments.

To support our miniature vacuum pump design efforts, Creare has developed statistical models of molecular drag pumps (MDP) in the free molecular flow regime. In this method, individual molecular trajectories through a simplified three-dimensional representation of the pump are calculated. The initial positions and velocities of the particles as they enter the pump are randomly generated, with statistics consistent with the gas states at the inlet and outlet of the pump. The free-molecular statistical simulation can be used to determine the probability that a molecule entering the pump at the inlet (outlet) exits through the outlet (inlet). In the free-molecular regime, these probabilities are sufficient to determine the pump's capabilities for compression, flow rate, etc.

We will describe the modeling methods, the verification of the models using previously published data, and the results of special experiments performed to verify that the models can be used to support new miniature pump designs.

11:40am **VT+MN-MoM11 Improvements in the Performance of Turbomolecular Pumps Beyond the Molecular Range**, *A. Chew, B. Brewster, I. Olsen, S. Ormrod*, Edwards Ltd, UK

A new range of turbomolecular pumps, nEXT, has been developed. This incorporates a new damping mechanism and pumping stage options. A new Siegbahn drag stage in combination with a regenerative mechanism are described in their combination with pure turbomolecular stages. Consequent increased backing pressures, high compression ratios and the facilitation of a boost port being used to back other turbos will be described.

# 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**

Two inter-related areas of rapid growth in mass spectrometry are i) Ambient ionization and ii) miniature mass spectrometers. This talk covers both topics. It attempts to provide the conceptual basis for these developments as well as summarizing the state of the technology and citing typical applications.

The authors believe that mass spectrometers are in the early stages of a radical transformation that will make them much more versatile and much more widely applied in situ than in the laboratory in future. At the same time they will increasingly be operated by non-technical staff including nurses, production line workers, industrial hygiene and food safety inspectors, surgeons and others who are not skilled in scientific instrumentation. The essential features needed for these developments are already in place, namely the rapid growth of regulatory and other demands for chemical analysis and the laborious nature of current standard laboratory methodology in many areas of chemical analysis.

Miniature and micro mass spectrometers are of great current interest. For many reasons, quadrupole ion traps are the most appropriately miniaturized mass analyzers and both microscale (10's of microns) and miniscale (100's of microns) instruments have been used. Arrays of such analyzers have further advantages in terms of reduction in power requirements. Most attention has gone to the mass analyzer but full systems (sampling, ion source, data capture and reduction) have been built and will be discussed, including a series of Mini mass spectrometers built in our labs. These handheld mass spectrometers have good performance (unit mass resolution to  $m/z$  500) and allow gases, solutions and surfaces to be analyzed using a variety of ionization methods including internal electron impact and external electrospray ionization and desorption electrospray ionization (DSI). These systems are pressure-tolerant, they provide tandem mass spectrometry capabilities and satisfies critical size and weight criteria while providing essentially instantaneous chemical analysis.

Ambient ionization - in which samples are examined without preparation in their native state is readily applied with miniature mass spectrometers. These methods, of which DESI is the prototypical example, do not require sample preparation and operate in the ambient environment. High throughput analysis of complex mixtures - with tandem mass spectrometry being used to resolve the overlapping chemical signatures - is possible. Examples range from benzene vapor in urban air to agrochemical residues in produce to phospholipid distributions in diseased tissue.

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.

Sandia National Laboratories is a multi-program laboratory operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin company, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

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 to 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. Volloero*, 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. Intrinsically, 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.

# Tuesday Morning, October 19, 2010

## Vacuum Technology

Room: Laguna - Session VT+MS-TuM

### Outgassing, Contamination Control, and Process Modeling

Moderator: M. Wuest, INFICON, Liechtenstein

8:00am **VT+MS-TuM1 Reduction of Hydrogen Content in Stainless Steel Vacuum Components**, *L.L. Wang, R.Y. Weinberg, K.A. Lao*, Los Alamos National Laboratory

Hydrogen is dissolved in stainless steel during the initial phases of production and fabrication. At room temperature, the dissolved hydrogen slowly diffuses out of the stainless steel. For stainless steel vessels assembled from commercially available vacuum components, we consistently measured constant rates of gas pressure increase in these sealed stainless steel vessels after they had been evacuated to  $1 \times 10^{-7}$  torr. The pressure in a 97 cc stainless steel vessel can reach up to 0.8 torr in six months at room temperature. The gas accumulated in these vessels, previously vacuum baked at 150°C for 48 hours to remove adsorbed gas, was analyzed to be essentially hydrogen. To determine how effective high-temperature vacuum bake out is in reducing the hydrogen content in the stainless steel components, we undertook a study that involved vacuum bakeout of the components at 400°C for 10 days and analysis of the hydrogen contents of the components with and without the vacuum bakeout. The hydrogen concentrations were measured by a LECO analyzer. The results will be presented and compared with that predicted by the Fick's law of diffusion.

8:20am **VT+MS-TuM2 Hydrogen Outgassing in a Small Vacuum Chamber**, *R.F. Berg*, National Institute of Standards and Technology

In a closed vacuum chamber, the problem of hydrogen outgassing from stainless steel increases with both the temperature and the chamber's surface-to-volume ratio. This talk will describe the outgassing in a chamber that is used to measure the vapor pressures of organic compounds in the range from 1 Pa to 100 kPa. The chamber, which is a small manifold built from stainless steel fittings and two capacitance diaphragm gauges, has a combination of challenges not usually present in a larger apparatus at room temperature. (1) Its volume of only 29 cm<sup>3</sup> created a relatively large surface-to-volume ratio. (2) Operating at temperatures as high as 200 °C greatly increased the outgassing rate. (3) The pressure gauges limited the maximum allowed bakeout temperature.

Closing the valve to the vacuum pump caused the pressure to increase nonlinearly with time. The initial rate slowed during several hours and usually became linear with time within one day. Intermittent pumping during one month at 200 °C showed that the linear rate decreased with an exponential time constant of approximately 11 days, which was consistent with the diffusion of hydrogen from the stainless steel fittings. Understanding this behavior is important because a pressure increase of 1 Pa/day ( $3 \times 10^{-10}$  Pa m<sup>3</sup>/s) can cause a significant error in the vapor pressure measurement. A model that accounts for the diffusion of hydrogen in the chamber wall and its nonlinear accumulation in the chamber volume will be compared to the pressure measurements.

8:40am **VT+MS-TuM3 Point-of-Use Abatement Devices and Exhaust Management Strategies**, *M. Sherer*, Sherer Consulting Services, Inc. **INVITED**

Semiconductor processes emit various contaminants which require exhaust management and in some cases point-of-use (POU) abatement. It is important to understand process exhaust management strategies, and to select the best, lowest cost-of-ownership POU abatement devices. This presentation will discuss these topics and provide relevant technical information.

9:20am **VT+MS-TuM5 Novel Instrument Capable of Efficient Gas Exchange to Remove Gas-phase Contamination in Complex Volumes Without Purging or High Vacuum**, *J. Brown, J. Hochrein, S. Thornberg*, Sandia National Laboratories

Countless systems used in research and in industry contain complex assemblies that are sealed in some type of enclosure, meant to isolate them from the harsh operating environment of the open atmosphere and to maintain a pristine internal atmosphere. Unfortunately, the internal atmosphere of any sealed component or system is, in the long-term, only as clean as the materials sealed within its enclosure. Over time, moisture or other volatile contaminants initially trapped in the materials can begin to

evolve and accumulate with potentially detrimental effects on the functionality of the component. This problem can be extremely difficult to address, depending on the physical and mechanical constraints of the particular system. Recently, an instrument was developed at Sandia National Laboratories that can "clean" the internal atmosphere of a critical optical component that cannot be subjected to conventional conditioning methods (such as N<sub>2</sub>/Ar purge, high-vacuum pumpdown, etc.). By using multiple pressurization and evacuation cycles tightly controlled within a narrow  $\pm 2$  psig window, the instrument fully and efficiently exchanges the liters of moisture- and contamination-laden internal gas of the component with clean, dry N<sub>2</sub>. This process is repeated as moisture from the internal materials diffuses back into the gas phase until, over time, the source of the moisture is depleted. This instrument has been successful in reducing the equilibrium gas-phase moisture levels in the optical component from the thousands of PPMv (parts per million by volume) to single-digit PPMv. This instrument, called the "Automated Pressure Cycler," will be discussed in detail.

10:40am **VT+MS-TuM9 Modeling, Design, Fabrication, and Characterization of a Pulsed Vacuum System**, *Z.C. Leseman, J. Butner*, University of New Mexico

Systems utilizing low to medium vacuum levels are becoming increasingly popular due to packaging of micro and nanoelectronic devices, exploration of surface phenomena, and gas-phase etching of materials. In this work, pulsed vacuum systems are modeled, designed, fabricated, and characterized. Modeling efforts focus on methods for calibration of volumes, pump-down / pressure-up times, and vacuum system configuration considerations. As a result of this systems of linear equations are developed and solved, as well as systems of coupled differential equations which are solved analytically and numerically (when necessary). As a result of this modeling effort a new method has emerged for vacuum processing at discrete pressures and discrete times. Experimental validation is presented in regards to specific applications: MEMS environmentally dependent stiction failure, vapor phase lubrication of MEMS, and XeF<sub>2</sub> vapor phase etching of Si.

11:00am **VT+MS-TuM10 Effects of Inlet Pipe Diameters on Pumping Performance of Turbomolecular Pump**, *F.-C. Hsieh, D.R. Liu, F.-Z. Chen*, National Applied Research Laboratories, Taiwan

The effects of inlet pipe diameters on pumping performance of turbomolecular pump (TP) are evaluated by commercial software VacTran. The result indicates that at inlet pipe diameter (D<sub>i</sub>) of 0.25 m the delivered pumping speed (DPS) of TP decreases from peak value (PV) of 680 L/s to about 430 L/s. However, DPS approaches to PV as D<sub>i</sub> increases to the largest one (1.0 m). Besides, the conductance of TP increases when D<sub>i</sub> increases. The conductance is proportional to the pipe radius at molecular flow. The pumping speeds (PS) versus the inlet pressure of TP and foreline (scroll pump) are evaluated. The PS for TP is higher than those obtained with foreline. Also, the DPS of TP is tested on an evaluation system constructed according to ISO-5302 standard. Good agreement between analysis and experimental data are shown. Finally, the throughput versus the inlet pressure of TP reveals linear trend in log-log scale within pressure range from  $3.1 \times 10^{-5}$  to  $9.3 \times 10^{-1}$  torr.

11:20am **VT+MS-TuM11 Optimal Configuration of a Radiometer Array for Low Pressure Applications**, *B. Cornella, A. Ketsdever*, University of Colorado at Colorado Springs, *N. Gimelshein, S. Gimelshein*, University of Southern California

A thin vane with a temperature gradient immersed in a rarefied gas will experience a force which tends to move the vane from the hot to the cold side. The radiometric force, as it is called, is the force that drives the Crookes radiometer. Applications of radiometric forces have been limited to date to high-density microdevices, most notably the atomic force microscope. However, applications can also involve larger devices in the low pressure regime (same equivalent Knudsen number). For an example, radiometric forces can act as a propulsion system to compensate disturbing forces on a vehicle traveling high in the atmosphere. Recent studies have shed new light on the relative influence of bulk radiometer area versus edge on force production, indicating that these effects are on the same order of magnitude in the Knudsen regime where the force is maximized (Kn=0.05). An experimental study has been conducted to investigate the impact of vane separation distance for a multiple-vane radiometer. This study is a first step in maximizing the force per unit volume (or mass) by optimizing the area versus edge geometries. Furthermore, this study provides experimental validation for today's numerical models involving rarefied radiometric flows. To emulate a near space environment, a 39" diameter vacuum chamber was used to set a range of pressures for the experiment from 0.1 to

10 Pa (corresponding to Knudsen numbers of 1.3 to 0.01). The experiment measures the total force of a one by three array radiometer configuration and compares it to a single vane with the same active area. Each individual radiometer vane consisted of a 40 mm square Peltier thermoelectric cooler where the temperature difference across the two surfaces was actively maintained at approximately 25 K. The relative separation between the vanes was varied from 0% (single vane setup) to 100% of the size of the individual vane element and the maximum forces between these varying configurations compared. Preliminary experimental results suggest that the total force produced by the overall radiometer increases with gap distance. Numerical results suggest that the optimum separation distance for maximum force production is around 75% of the vane height.

# Tuesday Afternoon Poster Sessions

## Vacuum Technology

Room: Southwest Exhibit Hall - Session VT-TuP

### Vacuum Technology Poster Session and Student Posters

**VT-TuP1 Vacuum Pressure Simulation for the Hard X-Ray Insertion Device Beamline 17A at NSLS, J.-P. Hu,** Brookhaven National Laboratory

Built in the 1980's, the insertion device beamline-17 at the X-ray storage ring of the Brookhaven Lab's National Synchrotron Light Source (NSLS) has been using superconducting-wiggler generated hard X-rays to facilitate cutting edge research. By sharing the wiggler's horizontal beam fan, three inline and one adjacent beamlines (17B1-B3 and 17C) have been designed to perform material stress-strain mapping, mineral phase transition under high-pressure, laser heating, and diffraction crystallography. To meet present-day high demand of hard X-rays for nano-structure probing via surface and interface scattering experiments and for large-volume high-pressure studies, a new beamline dubbed 17A, which also shares the wiggler's beam fan, has been constructed at immediately downstream to the common monochromator for all the branch beamlines at 17. For the purpose of improving beam quality, user safety and system vacuum, the degraded monochromator was replaced during the 17A construction by a custom-made monolithic unit to accommodate (1) a Si-crystal for the white beam bending (7.6-deg) into the 17A line, (2) a water-cooled white-beam filter followed by a collimated aperture for beam steering, (3) a Hevi-Met alloy of tungsten for bremsstrahlung shielding, (4) an ASME-certified burst disk for high pressure release, and (5) a sputter ion pump for outgas removal and high vacuum upkeep. Flanged to the beam exit port at the SS monochromator chamber is a round SS spool piece and a copper-brazed Be-window, installed to separate the beamline-17A vacuum from its upstream beamline-17 vacuum. At 1.4-meter downstream of the Si-crystal in monochromator is a 6-way cross, set to install a phosphor screen and a CCTV for the beam image viewing and profile recording. Along the beam path of 2.6-meter from the 6-port optical enclosure, a 200 L/s sputter ion pump is hooked and sealed beneath the round beam pipe to remove desorbed gases from photon-stimulated scattering amid two Be-windows. For beam size confinement, residual gas analysis, synchrotron radiation blockage and shock wave monitoring, a tungsten slit, a tee-port, a tantalum-plated safety shutter and a Be-window are respectively installed at 0.7-, 1.5-, 1.8- and 2.5-meter off the ion pump. Prediction of pressure profile along the 17A was performed using the Monte-Carlo based Molflow code for gas conductance estimate and the finite-difference based Vaccalc code for pressure distribution calculations. Details of beamline vacuum versus pre-cleaned and pre-baked assemblies encompassing the segmented beampipe will be presented. (Work performed under auspices of the US DOE, under contract DE-AC02-98CH10886)

**VT-TuP2 Yttria/ Rhenium Alloy Emission Filaments for Analytical Instrumentation, J. Manura, R. Shomo, C. Baker,** Scientific Instrument Services

Historically filaments used in most scientific instruments have been constructed from pure Rhenium. Rhenium has been the preferred material due to its resistance to oxidation and good emission qualities. However, Rhenium tends to be soft and has a tendency to warp and change shape during its operation. A new Yttria/Rhenium alloy has been developed for the purpose of improving the performance of filaments used in analytical instrumentation. The Yttria/Rhenium alloy filament exhibits the same electrical properties as pure Rhenium but has the advantage of not warping or changing shape, thereby improving the performance and lifetime of filaments used in analytical instrumentation.

Yttria alloys of rhenium were formed by sintering various concentration of yttria into rhenium. The sintered yttria/rhenium alloy bars were then drawn down to wires with diameters between 0.010" and 0.003".

Pure Rhenium filaments and Rhenium/Yttria alloy filaments of different configurations were tested to compare their properties. Testing was done using a custom filament station to measure the various filament electrical characteristics. Filaments were also tested in commercial instruments to monitor their performance. Electron microscopy studies were performed to study the grain characteristics.

Electrical studies in the filament testing station on pure rhenium filaments and various Yttria/Rhenium alloy filaments demonstrated that Yttria/Rhenium alloy filaments exhibited similar electrical properties as Rhenium. This enables these new alloy filaments to be used interchangeably with the standard rhenium filaments in analytical instrumentation.

Studies on the rhenium/yttria alloy filaments in commercial instruments demonstrated increased cycle lifetime as compared to standard rhenium filaments. The enhanced lifetime was attributed to the improved structural strength of the Yttria/Rhenium alloy filament. The Yttria/Rhenium filaments manufactured into different configurations all demonstrated less tendency to sag, warp or change shape as compared to pure Rhenium filaments.

Electron microscopy studies demonstrated that yttria oxide particles intermixed with the rhenium particles which minimized the grain growth in the alloy filament. In comparison, the pure rhenium filaments exhibited larger grain sizes. This smaller grain size in the alloy filament appears to strengthen the filament wire to provide a more stable filament that displays less sag or warping than pure rhenium filaments. The property of holding its shape has been demonstrated for multiple configurations. The Yttria/Rhenium material improves the performance of emission filaments used in analytical instrumentation.

**VT-TuP3 Calibration of Ultra-High Vacuum Gauge from  $10^9$  Pa to  $10^5$  Pa by Two-Stage Flow-Dividing System, H. Yoshida, K. Arai, M. Hirata, H. Akimichi,** National Institute of Advanced Industrial Science and Technology (AIST), Japan

A new two-stage flow dividing system has been developed for the calibration of ultrahigh vacuum gauges from  $10^9$  Pa to  $10^5$  Pa for  $N_2$ , Ar, and  $H_2$ . This system is designed based on the techniques of the calibration system in high vacuum region from  $10^{-7}$  Pa to  $10^{-2}$  Pa [1].

The system consists of four chambers: an initial chamber  $V_0$ , a flow divider  $V_1$ , a calibration chamber  $V_2$ , and an evacuation chamber  $V_3$ . Chambers between  $V_0$  and  $V_1$  and chambers between  $V_1$  and  $V_2$  are connected to each other with a capillary and a sintered filter, respectively. The chamber  $V_2$  is evacuated by a turbo molecular pump (1100 L/s for  $N_2$ ) through an orifice of 30 mm in diameter. The flow divider  $V_1$  is evacuated by a subsidiary turbo molecular pump (220 L/s for  $N_2$ ). The pressure  $P_0$  in the initial chamber is changed in 12 steps using a pressure controller in the range from  $10^2$  Pa to  $10^5$  Pa. The time interval for each step is 600 seconds. Following the change in the  $P_0$ , the pressure  $P_1$  in the flow divider and the pressure  $P_2$  in the calibration chamber similarly change from  $10^4$  Pa to 10 Pa and from  $10^9$  Pa to  $10^5$  Pa, respectively. The pressure  $P_2$  is determined from the pressure  $P_1$  using a pressure ratio of  $P_2$  to  $P_1$ . The ratio is pressure independent because the conductances of sintered filter  $C_1$  and the effective pumping speed of the turbo molecular pump though the orifice  $C_{main}$  are pressure independent at molecular flow region.

The modifications of this system from the previous one are listed below. (1) TiN coated stainless steel vacuum chambers are used as  $V_2$  and  $V_3$  to decrease outgassing from the chambers [2]. (2) The conductance of the sintered filter is 1000 times smaller than that of previous system to control the pressure in the range from  $10^9$  Pa to  $10^5$  Pa. (3) The ratio  $P_2/P_1$  is measured using a calibrated ionization gauge and a calibrated spinning rotor gauge. The ratio for  $N_2$ , Ar, and  $H_2$  is obtained to be  $6.41 \times 10^{-7}$ ,  $6.26 \times 10^{-7}$ , and  $8.36 \times 10^{-7}$ , respectively.

The pressure  $P_2$  is measured by an Extractor gauge (EXG) and an Axial-Symmetric Transmission gauge (ATG). The typical background pressure was  $(2-4) \times 10^{-9}$  Pa. These gauges were calibrated from  $10^9$  to  $10^5$  Pa for  $N_2$ , Ar, and  $H_2$  with an uncertainty of about 5% with the confidence level of 95% ( $k=2$ ). The linearities of these gauges were within  $\pm 2\%$ . The fluctuations of pressure indications were within  $\pm 2\%$  for 1 hour.

[1] H. Yoshida, K. Arai, H. Akimichi, M. Hirata, J. Vac. Sci. Technol. A 26 128 (2008)

[2] H. Akimichi, M Hirata, Metrologia 42 S184 (2005)

**VT-TuP4 Simultaneous Measurement of Pressure and Viscosity with a Resonant Sensor in a Viscous Flowing Gas, A. Kurokawa,** AIST, Japan, H. Hojo, T. Kobayashi, VPI Co., Japan

With a quartz tuning-fork resonator vibrating at the resonant frequency in the viscous flowing gas, we found that the measurement of the resonator's  $\Delta f$  and  $\Delta Z$  enabled to derive the pressure and the viscosity of the viscous flowing gas simultaneously. The parameter of  $\Delta f$  is the frequency change from its vibrating frequency in high vacuum. Another parameter of  $\Delta Z$  is the impedance change from the resonator impedance in high vacuum. Also the  $\Delta Z$  is related to the pressure and the viscosity of the gas. We focused on the pressure dependence of  $\Delta f$  and of  $\Delta Z$  to derive the pressure and the viscosity.

In this experiment, to achieve the precise measurements of  $\Delta f$  and  $\Delta Z$ , we paid careful attention to the temperature control because  $\Delta f$  was very sensitive to the temperature. We used the constant-temperature chamber in which the resonator, the driving circuit for the resonator, mass flow

controllers, and the absolute pressure gauge were installed. The temperature variation was  $\pm 0.1^\circ\text{C}$  during the experiment. In addition the driving circuit was stored in a thermostatic box which temperature was maintained at  $30\pm 0.02^\circ\text{C}$  to minimize the frequency drift. The driving circuit applied constant driving voltage ( $V_d$ ) to the resonator and the driving current ( $I_d$ ) passing through the resonator was monitored. The impedance of the resonator ( $Z$ ) was given by the ratio of  $V_d$  to  $I_d$ . The resonator was a tuning-fork type quartz resonator and had a vibration frequency of 32kHz. The measured gases were Ne, Ar,  $\text{N}_2$ ,  $\text{O}_2$ , Kr. The gas was charged at 130 kPa initially, and was vacuumed at the rate of 20 Pa/sec. The pressure of the gas was measured with the capacitance manometer.

The results showed that  $P-\Delta Z$  for every gas showed the same characteristics; the  $\Delta Z$  has larger value for higher pressure. For the higher mass of the gas showed the larger  $\Delta Z$  at atmospheric pressure except for Ne. The every  $P-\Delta Z$  curve did not cross each other except for Ne.

The  $P-\Delta f$  graph showed also the same tendency. The  $\Delta f$  has larger value for higher pressure, however, for the higher mass of the gas showed the larger  $\Delta f$  at atmospheric pressure including Ne. The every  $P-\Delta Z$  curve did not intersect one another except for Ne. Then showed close but not the same characteristics.

The  $\Delta Z-\Delta f$  plot revealed the difference between the  $P-\Delta Z$  and  $P-\Delta f$ . The  $\Delta Z-\Delta f$  curves did not intersect one another above 1 kPa and that the  $\Delta Z-\Delta f$  curves were arranged in order of the viscosity of the gas. Then the pressure and the viscosity of the gas can be derived simultaneously from  $\Delta Z-\Delta f$  curve.

**VT-TuP5 Study on Calibration Methods of Discharge Coefficient of Sonic Nozzles using Constant Volume Flow Meter, W.S. Cheung, J.H. Shin, S.B. Kang, K.A. Park, J.Y. Lim, KRISS, Republic of Korea**

This paper address technical issues in calibrating discharge coefficients of sonic nozzles used to measure the volume flow rate of low vacuum dry pumps. The first challenging issue comes from the technical limit that their calibration results available from the flow measurement standard laboratories do not fully cover the low vacuum measurement range of  $10^{-3} \sim 10^2$  mbar although the use of sonic nozzles for precision measurement of gas flow has been well established in national metrology institutes. The second one is to make an ultra low flow sonic nozzle sufficient to measure the throughput range of  $10^{-2}$  mbar-l/s. Those small-sized sonic nozzles exploited in this study not only to achieve the noble stability and repeatability of gas flow but also to minimize effects of the fluctuation of down stream pressures for the measurement of the volume flow rate of vacuum pumps. These distinctive properties of sonic nozzles are exploited to measure the pumping speed of low vacuum dry pumps widely used in the vacuum-related academic and industrial sectors.

Sonic nozzles have been standard devices for measurement of steady state gas flow, as recommended in ISO 9300. This paper introduces two small-sized sonic nozzles of diameter 0.03 mm and 0.2 mm precisely machined according to ISO 9300. The constant volume flow meter (CVFM) readily set up in the Vacuum center of KRISS was used to calibrate the discharge coefficients of the machined nozzles. The calibration results were shown to determine them within the 3 % measurement uncertainty. Calibrated sonic nozzles were found to be applicable for precision measurement of steady state gas flow in the vacuum process in the ranges of  $0.6 \sim 2,050$  cc/min. Those flow conditions are equivalent to the very fine gas flow with Reynolds numbers of  $26 \sim 8,500$ . Those encouraging results may confirm that calibrated sonic nozzles enable precision measurement of extremely low gas flow encountered very often in the low vacuum processes. Both calibrated sonic nozzles are demonstrated to provide the precision measurement of the volume flow rate of the dry vacuum pump within one percent difference in reference to CVFM. Calibrated sonic nozzles are applied to a new 'in-situ and in-field' equipment designed to measure the volume flow rate of low vacuum dry pumps in the semiconductor and flat display processes.

**VT-TuP6 High-k Gate Dielectric and Electrical and Surface Studies of  $\text{Al}_2\text{O}_3$ ,  $\text{HfO}_2$ ,  $\text{La}_2\text{O}_3$ ,  $\text{Al}_x\text{Hf}_y\text{O}_z$ , and  $\text{ZrO}_2/\text{HfO}_2$  on Silicon via Atomic Layer Deposition, G. Hernandez, R. Candler, S. Franz, Y.S. Lin\*, UCLA**

As the dimensions of the metal oxide semiconductor transistor shrink, quantum mechanical effects become more prominent. We are quickly reaching the limitations of  $\text{SiO}_2$  thickness of 10 -12 Å in which the tunneling current degrades device performance. Therefore, high dielectric constant materials are needed to replace  $\text{SiO}_2$  as the gate dielectric as we proceed to smaller devices. High-dielectric materials we study are  $\text{Al}_2\text{O}_3$ ,  $\text{HfO}_2$ ,  $\text{La}_2\text{O}_3$ ,  $\text{Al}_x\text{Hf}_y\text{O}_z$ , and  $\text{ZrO}_2/\text{HfO}_2$  deposited via Atomic Layer Deposit. We expect the aforementioned materials to have a lower leakage current and a band gap close to  $\text{SiO}_2$ . We expect the nanolaminates to have a

smoother interfaces as the expense of a band-gap in between each of its constituents. In order to characterize the electrical properties of each oxide, capacitors will be fabricated with oxide thicknesses of 50,100, and 150 Å. We will study the effects of different annealing /deposition temperature at the silicon-oxide interface by TEM. We also characterize the films with the material by XPS, AFM, and spectroscopic ellipsometry. The electrical properties will be determined by C-V and I-V measurements.

**VT-TuP7 Overview of Anharmonic Resonant Ion Trap Mass Spectrometry Technology, P. Acomb, G. Brucker, K. Van Antwerp, M.N. Schott, Brooks Automation, Inc.**

The poster will present the basics of an economical and commercially available mass spectrometer based upon Anharmonic Resonant Ion Trap Technology for mass selection. The mass separation method using electrostatic fields for ion trapping and the property of Autoresonance for mass selection will be shown. The key elements of gauge biasing, gas ionization, low-power RF-based mass separation and ion detection will be highlighted. Key performance characteristics of the anharmonic resonant ion trap will be summarized.

**VT-TuP8 Elements of Vacuum Quality Measurement using an Anharmonic Resonant Ion Trap Mass Spectrometry Technology, L. Landman, Brooks Automation, Inc.**

The poster will present the basics of a Vacuum Quality Measurement System using the inputs from commercially available mass spectrometer based upon Anharmonic Resonant Ion Trap Technology for mass selection, a total pressure gauge, external signals and a scripting tool to transform complex measurements into a single valued output.

**VT-TuP9 A Computationally Simple, Wafer-to-Feature-Level Model of Etch Rate Variation in Deep Reactive Ion Etching, J.O. Diaz\*, H.K. Taylor, Massachusetts Institute of Technology, R.J. Shul, R.L. Jarecki, T.M. Bauer, Sandia National Laboratories, D.S. Boning, Massachusetts Institute of Technology, D.L. Hetherington, Sandia National Laboratories**

Modeling etch rate variation in Deep Reactive Ion Etching (DRIE) helps to identify possible defects in MEMS and IC devices arising from sub-optimal etching depths and times. Besides tool-specific properties, such as the chamber design, another cause for the observed non-uniformity effects is the particular wafer pattern employed. At the wafer scale, previous studies have shown that wafers with a large percentage of open (exposed Si) area, or pattern density, exhibit a radial center-low etch-rate distribution, while those with low pattern density achieve radial center-high etch rates. At the die scale, it is widely known that etch rate decreases as local pattern density increases. Furthermore, at the feature scale, the microloading effect describes how adjacent features tend to compete for radical species, thus decreasing overall etch rates within individual features.

We present a model to capture these pattern-dependencies by tracking the spatial and temporal distribution of the ion and radical species within the DRIE chamber. The model implementation uses a time-stepped algorithm with three levels – corresponding to the three different length scales – and a coarse-grain approach where multiple features in a given region are characterized by a particular shape, size and density. The local radical species concentration distribution above the wafer is determined at each time step using current feature geometries to compute their Knudsen transport coefficient which is linked to the radical transport mechanisms within other areas in the chamber. At the end of each time step, etch rate estimates based on this radical concentration distribution and current feature geometries are used to update feature depth information for the next time interval. At the wafer scale, our modeling results achieve a success comparable to that of previously-developed wafer-level models with an etch rate RMS error percentage between 2.1% and 8.2%. The results also show that feature-level etch evolution substantially impacts the wafer-level fluorine concentration and thereby modifies the wafer and die etch rate uniformity. We expect a similar model could be incorporated into CAD software tools to evaluate masks and correct potential design issues before they are made. Our results also shed light on possible tool and process modifications to allow users the capability of altering across-wafer etch rate variability. Sandia National Laboratories is a multi program 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.

**VT-TuP10 Design, Simulation, and Implementation of Plasma Enhanced Atomic Layer Deposition in a Laminar Flow Reactor, K. Kellogg\*, P. Falvo, University of South Florida, S. Lee, University of South Florida, T. Wright, J. Wang, University of South Florida**

A plasma enhanced atomic layer deposition reactor (PE-ALD) was built for the purpose of growing thin films on wafers up to 2.5" in diameter. Internationally, papers have been published describing characteristics of

\* VT Student-Built Vacuum Systems Poster Competition

both homebuilt [1,2,3] and commercially available ALD reactors [4]. The construction of this reactor was strategically designed using these descriptions, within an allowable project time and budget. Design characteristics include an inert carrier gas, millisecond speed precursor valves, a remotely generated inductively coupled plasma, and a chamber with a high volume to surface ratio geometry. The reactor will act to complement and increase the current application repertoire versus our commercially available reactor located in the University's thin films laboratory. In this regard, the chamber must be optimized to accommodate unique recipe applications currently unattainable with the in-house system. The functionality of this reactor will include three separate modes of operation: a thermal reaction mode (thermal ALD) for use with general recipe applications, an isolated chamber mode necessary for high aspect ratio substrates, and a plasma enhanced mode (plasma enhanced ALD) for greater process recipe versatility such as metals and nitrides. ALD allows for a precision unattainable with other deposition processes. Unlike CVD, ALD is not dependent upon precursor flux upon the substrate surface, instead relying upon step-wise  $A + B = P$  synthesis. Reactor characteristics such as laminar gas flow and plasma ion locality concentration and intensity will be modeled with COMSOL finite element simulations. ALD deposition cycle times are optimized according to ALD chemical reactions and by in-situ monitoring of sample growth rates by means of fiber optic spectroscopy. Important considerations included an optimized pumping rate and a minimization of unavoidable deposition upon all surfaces other than the process wafer. Process optimization was also pursued by means of vacuum gauge feedback and automation of precursor valve cycle sequence by means of a Lab View enabled PC. Other automated controllable growth parameters include substrate heater temperatures, reactor wall temperatures and the energies of plasma ion bombardment upon the substrate surface species. Safety concerns have also been addressed by ensuring suitable gas exhaust, pump maintenance, hard-wired safety valve shut-off programming and gas cylinder and hazardous materials safety training of individual users. The chamber design, multitude of process optimizations, and comparisons with existing designs and models allow for substantial research parameters to be explored and discussed.

References: [1] J. W. Elam, M. D. Groner, and S. M. George, "Viscous Flow Reactor with Quartz Crystal Microbalance for Thin Film Growth by Atomic Layer Deposition," *Review of Scientific Instruments*, Vol. 73 No. 8, Aug. 2002, pp. 2981-2987 [2] H. C. M. Knoops, L. Baggetto, E. Langereis, M. C. M. van de Sanden, J. H. Klootwijk, F. Roozeboom, R. A. H. Niessen, P. H. L. Notten, and W. M. M. Kessels, "Deposition of TiN and TaN By Remote Plasma ALD for Cu and Li Diffusion Barrier Applications," *Journal of the Electrochemical Society*, Vol. 155, No. 12, Oct 2008, pp. G287-G294 [3] G. A. Ten Eyck, J. J. Senkevich, F. Tang, D. Liu, S. Pimanpang, T. Karaback, G. Wang, T. Lu, C. Jezewski, and W. A. Lanford, "Plasma-Assisted Atomic Layer Deposition of Palladium," *Chemical Vapor Deposition*, Vol 11, No. 1, 2005, pp. 60-66. [4] S. B. S. Heil, J. L. van Hemmen, C. J. Hodson, N. Singh, J. H. Klootwijk, F. Roozeboom, M. C. M. van de Sanden, and W. M. M. Kessels, "Deposition of TiN and HfO<sub>2</sub> in a Commercial 200mm Remote Plasma Atomic Layer Deposition Reactor," *Journal of Vacuum Science and Technology A*, Vol. 25, No. 5, Sept/Oct 2007, pp. 1357-1366.

**VT-TuP11 A Cryogenic Vacuum Chamber for Low Temperature Thermophotovoltaic Testing.** *D. DeMeo\*, T. Vandervelde*, Tufts University

Thermophotovoltaics (TPV) are devices capable of converting infrared electromagnetic radiation into electricity. Strained Layer Superlattices allow TPV devices to operate at longer wavelengths. In order to determine the performance of these devices, a unique test apparatus was designed and constructed. As the devices become sensitive to longer wavelengths (lower temperatures) in the infrared, the need to control the sample's ambient temperature becomes paramount. Here, we present a custom, cryogenic vacuum chamber specifically designed to test long wavelength TPV cells. The tester utilizes two copper heat shields cooled via conduction with two liquid nitrogen reservoirs to block outside thermal radiation. A blackbody source illuminates a temperature controlled sample at high vacuum,  $\sim 10^{-6}$  Torr. The chamber is also connected to multiple thermocouples and a source-meter for measurement and testing purposes. This test apparatus will enable future research into low temperature TPV and other optoelectronic devices.

**VT-TuP12 Experimental Approach to Equalizing the Orifice Method with the Throughput One for the Measurement of TMP Pumping Speed.** *J.Y. Lim, S.B. Kang, J.H. Shin*, Korea Research Institute of Standards and Sciences, Republic of Korea, *D.J. Cha*, Kunsan National University, Republic of Korea, *D.Y. Koh*, Korea Institute of Machinery and Materials, Republic of Korea, *W.S. Cheung*, Korea Research Institute of Standards and Sciences, Republic of Korea

Methods of the characteristics evaluation of turbo-molecular pumps (TMP) are well-defined in the international measurement standards such as ISO, PNEUROP, DIN, JIS, and AVS. The Vacuum Center in the Korea Research Institute of Standards and Science (KRISS) has recently designed, constructed, and established the integrated characteristics evaluation system of TMPs based on the international documents by continuously pursuing and acquiring the reliable international credibility through measurement perfection.

The measurement of TMP pumping speed is normally performed with the throughput and orifice methods dependent on the mass flow regions. However, in the UHV range of the molecular flow region, the high uncertainties of the gauges, mass flow rates, and conductance are too critical to precisely accumulate reliable data. With UHV gauges of uncertainties less than 15% and a calculated conductance of the orifice, about 35% of pumping speed uncertainties are experimentally derived in the pressure range of less than  $10^{-6}$  mbar. In order to solve the uncertainty problems of pumping speeds in the UHV range, we introduced an SRG with 1% accuracy and a constant volume flow meter (CVFM) to measure the finite mass flow rates down to  $10^{-3}$  mbar-L/s with 3% uncertainty for the throughput method. In this way we have performed the measurement of pumping speed down to less than  $10^{-6}$  mbar with an uncertainty of 6% for a 1000 L/s TMP. In this article we suggest that the CVFM has an ability to measure the conductance of the orifice experimentally with flowing the known mass through the orifice chambers, so that we may overcome the discontinuity problem encountering during introducing two measurement methods in one pumping speed evaluation sequence.

**VT-TuP13 Implementation of a Lambertson Magnet in an Ultrahigh Vacuum Electron Storage Ring.** *V. Anferov, J. Doskow, G. East, S.Y. Lee, T. Rinckel, C. Romel, T. Sloan, P. Sokol*, Indiana University

The Advanced Electron-Photon Facility (ALPHA), built at Indiana University, is a multipurpose electron accelerator to be used for DoD radiation effects testing as well as IU's interest in a compact high-brightness x-ray source. ALPHA consists of a 50 MeV linear accelerator source and 20 m storage ring which operates at  $1 \times 10^{11}$  Torr. A Lambertson magnet, used to inject/extract the electron beam into and out of the ring, has been uniquely designed for optimal vacuum behavior and septum straightness while maintaining magnetic field quality. The design minimizes the ultrasonically-tested, 1018 steel pole tip exposure to UHV via a nickel plated surface and an exterior stainless steel vacuum body, welded to the pole tip. The magnet assembly yielded positive results in magnetic field and vacuum testing and is currently being commissioned in the ring.

# Wednesday Morning, October 20, 2010

## Vacuum Technology

Room: Laguna - Session VT-WeM

### Accelerators, Large Vacuum Systems, and Vacuum Surfaces

**Moderator:** R.F. Berg, National Institute of Standards and Technology

8:00am **VT-WeM1 Architecture and Operation of the Z Pulsed Power Facility Vacuum System, J.W. Weed, Sandia National Laboratories, D.W. Petmecky, Ktech Corporation, A.C. Riddle, Sandia National Laboratories**  
**INVITED**

The Z Pulsed Power Facility at Sandia National Laboratories in Albuquerque, New Mexico, USA is one of the world's premier high energy density physics facilities. The Z Facility derives its name from the z-pinch phenomena which is a type of plasma confinement system that uses the electrical current in the plasma to generate a magnetic field that compresses it. Z refers to the direction of current flow, the z axis in a three dimensional Cartesian coordinate system. The multiterawatt, multimegajoule electrical pulse the Facility produces is 100-400 nanoseconds in time. Research and development programs currently being conducted on the Z Facility include inertial confinement fusion, dynamic material properties, laboratory astrophysics and radiation effects. The Z Facility vacuum system consists of two subsystems, center section and load diagnostics. Dry roughing pumps and cryogenic high vacuum pumps are used to evacuate the 40,000 liter, 200 square meter center section of the facility where the experimental load is located. Pumping times on the order of two hours are required to reduce the pressure from atmospheric to  $10^{-5}$  Torr. The center section is cycled from atmosphere to high vacuum for each experiment. The facility is capable of conducting one to two experiments per day. Numerous smaller vacuum pumping systems are used to evacuate load diagnostics. The megajoules of energy released during an experiment causes damage to the Facility that presents numerous challenges for reliable operation of the vacuum system.

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.

8:40am **VT-WeM3 Vacuum System for the Large-Scale Cryogenic Gravitational Wave Telescope (LCGT), R. Takahashi, National Astronomical Observatory of Japan, Y. Saito, T. Suzuki, KEK-High Energy Accelerator Research Organization, Japan**

The large-scale cryogenic gravitational wave telescope (LCGT) project is proposed to open a new window for astronomy, which will be able to detect signals from the binary neutron star coalescence at 240Mpc away. LCGT requires an ultra-high vacuum tubes which the laser beams pass through. Two 3-km vacuum tubes are kept in  $\sim 10^{-7}$ Pa of vacuum pressure so as to reduce scattering-effects due to residual gas molecules.

Mirrors of the main interferometer are cooled to 20K to reduce thermal noises. The super-insulator (SI) which consists of multi-layered organic films is generally used for a thermal insulation of cryostat. However, the SI should not be applied to the cryostat of LCGT to avoid contamination on the extremely sensitive mirrors. We plan to use multi-layered metal shields with low emissivity and low outgassing for a thermal insulation.

9:00am **VT-WeM4 Working toward XHV: Characterization and Improvements of the Vacuum System for GaAs Photoemission Electron Sources, M.L. Stutzman, P.A. Adderley, A. Comer, M. Poelker, Jefferson Lab**

The operational lifetime of a DC electron source using GaAs photocathode material is limited primarily by the system vacuum; the residual gasses ionized by the electron beam are accelerated into the photocathode where they cause damage and limit photocathode yield. Though we operate in the deep-UHV range, improvements to the vacuum should increase lifetime for today's electron sources, and is essential for proposed future accelerators needing considerably higher average current. This talk describes our efforts to improve vacuum in the Jefferson Lab polarized electron source, including efforts to characterize NEG and ion pumps in the deep-UHV range, carefully determine the x-ray limit of our Leybold extractor gauges, and quantify the reduction in outgassing from stainless steel chambers after a long 400°C heat treatment. The goal of these studies is to determine which factors primarily limit our ultimate pressure, to find ways to lower the

ultimate pressure for future electron sources, and to quantify these improvements.

9:20am **VT-WeM5 The Role of Vacuum Technology in the Production of Neutron Generators, J.L. Provo, Sandia National Laboratories**

Neutron generators are neutron source devices which are composed of small linear accelerators that produce neutrons by fusing isotopes of hydrogen. Such devices were first used in the ignition of nuclear weapons but many commercial applications have been developed over the past 50 years. The critical component of a neutron generator is a small particle accelerator called a neutron tube. Neutron tubes are composed of several components which include an ion source, ion optic elements, and a target on to which ions are accelerated. All components are enclosed in a vacuum tube enclosure with a high voltage insulator between the ion source and the target. The ion source and accelerator high voltages are provided by external power supplies either of electronic design or of a ferroelectric explosive design. Electronic generators can be used after function testing while explosive generators are destroyed.

Neutron tubes thus are similar to vacuum tubes previously used in televisions, radios, etc., and processes used for production of these devices can also be used for neutron tubes. Because of their application, the quality requirements for manufacturing such devices for weapons are very rigorous and are of the highest quality standards. These devices typically are designed and fabricated to operate over a temperature range of -65 °F to +168°F with 99.99+% reliability at over 95% confidence. These are quality levels rarely found in any industry. To support these requirements, the latest in applied science and technology in analysis of materials and chemical and vacuum processes were utilized.

Described will be analyses and processes used in the characterization of materials and components used in vacuum neutron tube manufacture. These include surface analysis techniques used to prove materials have specified constituents with no impurities. Cleaning processes used to prepare tube components prior to sub-assembly, which include plasma cleaning, and vacuum firing, vacuum brazing assembly processes as well as thin film deposition processes for tube ion sources and targets, occluder film hydriding processes, and final tube exhaust processes, all of which use vacuum technology will be briefly described.

Some weapon and commercial versions of neutron tubes will also be described with external photos, their history, and their applications. It is very evident that the application of vacuum technology was absolutely necessary to produce the controlled environments that meet the quality standards for neutron tube weapon application as well as for the many commercial applications that require advanced-materials processing and manufacture in use now and in the future.

10:40am **VT-WeM9 Application of Electrochemical Buffing to Niobium Superconducting RF Cavity, S. Kato, M. Nishiwaki, P.V. Tyagi, S. Azuma, F. Yamamoto, KEK-High Energy Accelerator Research Organization, Japan**

Niobium electropolishing for SRF cavities are generally considered to be the best technology today.

However, hydrofluoric and sulfuric acid mixture usually used in the EP process is harmful and requires us carefully controlled handling of it and the many additional facilities. In this article, we propose a new application of electrochemical buffing onto niobium SRF cavity. In the method of electrochemical buffing, a rotating disk with abrasive fine particles where electrolyte is supplied is pressed against the workpiece. The disk and the work function as a cathode and an anode, respectively and an aqueous solution of sodium nitrate is used for the electrolyte. This technique brings us a couple of advantages like high etching rate, ultra small surface roughness, cost-effective and environment-compatible polishing.

11:00am **VT-WeM10 Development of UHV Field Emission Scanner for Surface Study of Niobium SRF Cavity, S. Kato, M. Nishiwaki, V. Chouhan, P.V. Tyagi, T. Noguchi, KEK-High Energy Accelerator Research Organization, Japan**

It is mandatory to investigate field emission on Nb SRF cavity systematically since strong field emission often limits the cavity performance. The field emission strength and the number of emission sites strongly depend on Nb surface properties which are determined by its surface treatment and handling. Field emission scanner developed allows us to measure a distribution of the field emitting sites over a sample surface at a given field strength along with its FE-SEM (field emission scanning electron microscope) observation and energy dispersive x-ray analysis. The field emission scanner consists of a sample stage driven by piezo actuators and an anode needle driven by precise 3D stepping motors. In addition, this

system was newly equipped with a sample load-lock system for existing UHV suitcases. The compact scanner was installed into the space between the object lens and the SEM sample holder. The UHV pumps were additionally installed in order to improve the base pressure down to UHV to reduce adsorbates during the measurement. This article describes development of the field emission scanner and its preliminary results of the application to niobium samples.

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