# Wednesday Afternoon, October 17, 2007

Vacuum Technology

### Room: 618 - Session VT-WeA

### Miniature, Portable and Space Vacuum Applications

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

# 1:40pm VT-WeA1 Vacuum Pumping Requirements for Miniature Mass Spectrometers, *R. Ellefson*, Consultant

A trend in mass spectrometers (MS) for gas analysis is miniaturization to occupy less volume when attached to vacuum chambers and additionally to decrease weight and power consumption for portable and space probe instruments. This presentation identifies the pumping system requirements for different MS applications. When sample impurities like H<sub>2</sub>, H<sub>2</sub>O, CO,  $N_2$ ,  $O_2$  and  $CO_2$  are being analyzed, a lower base pressure (e.g.  $<10^{-8}$  Torr) is required for low background ion currents from MS outgassing resulting in a low detection limits for these species in the sample. For analyzing hydrocarbons and other species with masses greater than 44, a higher base pressure (e.g. 10<sup>-6</sup> Torr) can be tolerated if the hydrocarbon background is kept low by initial cleaning and operating methods. Factors dictating the operating pressure of the MS and the resulting gas throughput are presented together with scaling rules for miniaturization. Given the pumping requirements, a high vacuum pump can be selected or adapted. A comparison is made of throughput and capture pumps for MS applications focusing on the end use of the MS as a process monitor or a field portable instrument. Examples from literature are given. Finally the pumping system for gas sampling and pressure reduction to the MS is addressed.

### 2:00pm VT-WeA2 Miniature High Vacuum Pump for Mars Analytical Instruments, R.J. Kline-Schoder, P.H. Sorensen, Creare Incorporated INVITED

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 mass spectrometers, and miniaturized versions of other analytical instruments are under development. However, the vacuum systems required to support these sensors remain large, heavy, and power hungry. In particular, high vacuum systems of adequate performance continue to be too large for systems such as time-of-flight, quadrupole, and ion trap mass spectrometers that are intended to be man-portable or to be deployed on UAVs, balloons, or interplanetary probes. The 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 10 years, Creare has been developing the technologies required to design and build miniature high vacuum pumps. During this time, we have designed and built two small high vacuum pumps that have the following pumping characteristics: a compression ratio for air that is greater than  $10^8$ ; a pumping speed of about 5 L/sec; and 10 W power consumption for an exhaust pressure of 10 Torr. The smallest of these pumps has a mass of 130 g, a diameter of 1.3 in., and an overall length of 2.3 in (i.e. the size of a c-cell battery). The slightly larger pump has a mass of 500 g, a diameter of 2.0 in., and an overall length of 4.6 in (i.e. the size of a soda can). The larger version is being space qualified for use on a NASA Mars mission scheduled for launch in 2009. The challenges of designing and building miniature turbomolecular/molecular drag pumps include: design of pump geometry in regions where little data exist, the need for precision machining of components, and the electromagnetic and mechanical design of very high speed, efficient, miniature electric motors. Data will be presented that show the performance, over a wide temperature range, of a brassboard prototype of the pump NASA currently plans to deploy on the Mars Science Laboratory mission.

#### 2:40pm VT-WeA4 Development of MEMS for Space Applications, *P.W. Valek, D.J. McComas*, Southwest Research Institute INVITED Space flight missions have critical requirements such as low mass, low power, and high reliability. The technology of Micro-Electro Mechanical Systems (MEMS) naturally has many properties that address these space flight requirements. MEMS devices are built using the same techniques that have been developed by the semiconductor industry so they share the same benefits that we have come to expect from modern electronics, i.e., reduced size, low mass, low cost, etc. While there has been significant research on how MEMS technology operates in the more "normal" environments encountered for consumer electronics and biological application, for the

benefits of MEMS technology to be fully realized for space applications their operation in a vacuum needs to be understood. The relative importance of different physical mechanisms shifts when going from a macro-scale world to that of the micron scale. For example, surface tension and stiction are easily dealt with on the macro scale but become significant problems on the MEMS scale. We will discuss the challenges and opportunities that are present for MEMS technology when used in space or any vacuum environment. For example, MEMS oscillators operating in vacuum have Q-values many orders of magnitude larger than when operated at atmospheric pressures.<sup>1</sup> We will present results from our testing of MEMS devices in a vacuum environment and discuss the implications for further space instrumentation development.

<sup>1</sup> McComas et al., "Space applications of microelectromechancial systems: Southwest Research Institute vacuum microprobe facility and initial vacuum test results", Rev. Sci. Inst., Vol 74, 2003.

4:00pm VT-WeA8 The Role of Vacuum-Based Processes in Developing High Performance Chemical Microsensors, S. Semancik, D.C. Meier, J.K. Evju, M.J. Carrier, C.B. Montgomery, National Institute of Standards and Technology, K. Newcomb, C.L. Keast, M.I.T. Lincoln Laboratory INVITED

There is a growing demand for solid state chemical microsensors that are capable of analyzing gas phase compositions encountered in a wide range of application areas, from process control to space exploration and health care. In certain cases these small sensing devices would be used instead of more expensive and cumbersome instrumentation, and in others they would enable chemical monitoring within dispersed multipoint networks which are not amenable to instrument-based measurements. While the necessary detection characteristics vary with application, the defense/homeland security sector provides what are arguably some of the most demanding performance requirements for such microsensors: rapid detection; sensitivities to hazards such as chemical warfare agents (CWAs) and toxic industrial chemicals (TICs) at nmol/mol (ppb) and even pmol/mol (ppt) concentrations; reliability and robustness to avoid false target readings in practical backgrounds; and, extended lifetimes. This presentation will focus on vacuum-based processing and vacuum-related phenomena that enable the fabrication and evaluation of MEMS-based, chemiresistive microsensor array devices being developed for detection of low level chemical hazards in air-based backgrounds (including interference compounds). Microarray device platforms (1000s of devices on 6 inch wafers) are fabricated at a silicon foundry through a multi-step processing schedule including nearly two dozen controlled vacuum procedures (e. g. - etching, CVD, PVD). The incorporation of nanostructured sensing materials onto the  $\sim 100~\mu m$ (microhotplate) array elements of our devices is achieved using a variety of methods, including thermally activated, self-lithographic CVD (at 3 Pa), and an ion etch pre-process (at  $\sim 10^{-5}$  Pa base pressure) has been shown to produce good sensing material contact to the microdevice electrodes, which is critical for attaining high sensitivity and reliable operation. In addition, vacuum phenomena come into play while evaluating the microarray sensors, since very low concentrations of target analytes (often with low vapor pressures) must be injected into air-based backgrounds and delivered to a device exposure point within our testing system. Technical aspects (enhanced analytical content for species recognition, redundant elements, etc.) that have allowed us to achieve sub-ppb CWA simulant detection with our microsensors will be discussed.

#### 4:40pm VT-WeA10 A Novel Electrostatic Ion Trap Mass Spectrometer, A.V. Ermakov, B.J. Hinch, Rutgers University

We have developed, built and tested several prototypes of a novel mass spectrometer which operates with an entirely new basic principle - i.e. using an electrostatic resonance ion trap. This mass spectrometer has an unlimited mass range, is capable of achieving higher sensitivity, and has much faster scan rates than the widely used (larger size and) more complicated quadrupole or magnetic sector mass spectrometers. In addition, the new mass spectrometer is very compact (less than 2" long), and requires very small power (in the mW range, excluding ionizer) as it uses only static potentials and a very small RF voltage (in the 100mV range). The high sensitivity of our mass spectrometer at low background pressure (below 10<sup>-8</sup> torr) allows for the possible construction of an easily portable analytical instrument (handheld, if necessary.) A portable system could use only a battery powered comp act ion pump, and would not require (noisy, bulky, and energy consuming) mechanical pumps.

# Thursday Morning, October 18, 2007

Vacuum Technology

### Room: 618 - Session VT-ThM

### Pumping, Pressure Measurement and Calibration

### Moderator: J. Luby, BOC Edwards

### 8:00am VT-ThM1 Review of Seven Years Field Application Experience of an EPX Single Mechanism for High Vacuum Pumping, A.D. Chew, C. Shaw, BOC Edwards, UK INVITED

A single dry pump mechanism capable of reaching high vacuum and itself exhausting to atmospheric pressure has been a "vacuum-technology panacea". The development and deployment of a single-shaft, high-speed EPX pump isince has gone some considerable way to achieving this goal. This paper will describe the stages in the development history and expanding applications the pumps has been applied to. This will be further illustrated by specific applications examples, reliability and economic experiences.

### 8:40am VT-ThM3 How to Efficiently Combine Ion Pumps and Getter-Palladium Thin Films, C. Paolini, M. Mura, F. Ravelli, Varian S.p.A., Italy

Non-evaporable getters (NEG) have been extensively studied in the last years for their sorption properties towards many gases. In particular, an innovative alloy produced in the form of thin films by magnetron sputtering was developed and characterized at the European Center of Nuclear Research (CERN). It is composed of Ti-Zr-V and protected by an overlayer of palladium (Pd), according to a technology for which we got the license. The use of NEG-Pd thin films in combination with ion getter pumps allows to obtain a simple and easy to handle pumping device for UHV and XHV applications. In order to show how it is possible to apply this coating technology to the internal surface of different types of ion pumps, several tests were carried out on pumps of various shape, size (in terms of nominal pumping speed) and type (diode, noble diode and triode). A special care was taken during the thermal cycle of bakeout and activation of the pumps, in order to preserve the internal film from the contamination due to the sputtering of the cathodes and/or from the interdiffusion of its components. Some important remarks about the most appropriate conditions of pressure and temperature will be discussed. The performances of the NEG-Pd coated ion pumps were evaluated in terms of ultimate pressure, nitrogen and hydrogen pumping speed. The contribution of the thin film is particularly relevant for the pumping of this last gas, due to its high sticking factor for palladium and to the great sorption capacity of the underlying getter. Finally, the possibility of further improvement of the performances by substituting the palladium with other Pd-based alloys will also be evaluated.

### 9:20am VT-ThM5 Vacuum Improvements and Characterizations for the Jefferson Lab Polarized Electron Source, *M.L. Stutzman*, *P.A. Adderley, J. Grames, M. Poelker*, Thomas Jefferson National Accelerator Facility

Improving vacuum is a necessary step toward improving photocathode lifetime in DC high voltage polarized electron sources, which is an important goal for both the CEBAF nuclear physics accelerator at Jefferson Lab and future facilities. A new load-locked photogun vacuum system has been constructed using many vacuum improvements, including vacuum firing and NEG coating the gun high voltage chamber. The vacuum characteristics of the new photogun are described in the context of traditional vacuum measurements but perhaps more importantly, a vacuum assessment is made by comparing new and old photogun performance.

#### 9:40am VT-ThM6 Theory and Design of a Pirani-style Thermal Conductivity Vacuum Gauge with Unique Geometries and Control Circuitry, P.C. Arnold, Brooks Automation, Inc.

Characteristics of Pirani-style gauges and their causes for inaccuracy due to errors in temperature compensation will be presented. The principles of sensor thermal end losses as they contribute to pressure indication errors as well as errors due to changes in ambient temperature and non-uniformities in mounting structures will be discussed with special attention to reducing those uncertainties. The design, called Conductron (R) technology, is found to have usable pressure indication up to an atmosphere without utilization of gas convection enhancement geometry which causes orientation dependency. A geometry and method of operating the gauge, departing from the conventional Wheatstone bridge, that avoids common pitfalls of conventional Pirani gauge operation will be described. Certain operational measurements will be shown to avoid the more difficult determinations of (a) power lost to gas conductance and (b) measurement of sensor environment temperature, often used in both transducing these data to an indicated pressure and also providing temperature compensation. Also shown will be a unique method for arriving at the indicated pressure from those operational measurements. A pressure range from the low mTorr to atmosphere is encompassed by this design.

10:00am VT-ThM7 Capillary Flow Meter for Calibrating Spinning Rotor Gauges, R.F. Berg, National Institute of Standards and Technology Below 1 Pa, the NIST Pressure & Vacuum Group generates known pressures by flowing gas through an orifice with a calculable impedance. The gas flow is a leak from a small volume held at a higher pressure. Slowly inserting a piston into the volume holds the volume's pressure constant, and the known insertion rate and cross section of the piston, plus the pressure and temperature of the volume, yield the gas flow rate. This talk will describe the performance of new gas flow source based on a capillary flow impedance. Knowing the input pressure, output pressure, and temperature of the capillary yields the gas flow rate through the capillary. The capillary flow meter uses large pressures (30 - 300 kPa) that can be accurately measured, it requires no moving parts aside from valves, and it provides a steady flow for days instead of minutes. The new flow meter comprises a coil of quartz capillary with an inner diameter of 0.1 mm and a commercial pressure gauge package. Its maximum flow rate of 0.2 micromol/s (about 0.2 standard cubic centimeter per minute) covers the range that is useful for calibrating spinning rotor gauges. The flow meter relies on a hydrodynamic model that was developed for NIST transfer standards for larger gas flows with a relative uncertainty better than 0.1 %. A preliminary comparison at 0.1 micromol/s showed agreement between the piston flow meter and the capillary flow meter to within 0.2 %. Comparisons at other flow rates and extension of the hydrodynamic model to handle exit pressures below 30 kPa will be discussed.

#### 10:20am VT-ThM8 A Non-Destructive Partial Pressure X-Ray Analysis Method for Kr and Xe Gas Filled Encapsulated Devices, *P.F. Somssich*, *K.J. Zuk*, Osram Sylvania

A method to non-destructively measure the gas fill pressure of glassencapsulated gas devices, e.g. lighting products will be described. The technique, first developed at GTE Laboratories in Waltham, MA, has recently been further expanded to include a wider range of devices (0.02cc and above) and pressures (15 Torr to 10 Atm.), all of which contain a xenon or krypton fill gas. When analysis results of an EDXRF instrument are combined with that of an absolute pressure-volume analyzer, calibration curves were generated allowing for subsequent non-destructive fill pressure determinations with an accuracy of approx. +/- 10%. The EDXRF analysis generates additional useful qualitative information which will also be presented, e.g. detecting the presence of iodine and other salts. Possible applications for 100% quality testing of products using a variant of the test, sub-second analysis, will be discussed.

# Thursday Afternoon, October 18, 2007

Vacuum Technology

### Room: 618 - Session VT1-ThA

# Adsorption/Desorption Phenomena on Vacuum Materials

Moderator: N. Peacock, MKS Instruments, Inc.

### 2:00pm VT1-ThA1 Surface Morphology and Surface Composition of Vacuum Fired Stainless Steel\*, *M. Leisch*, Graz University of Technology, Austria INVITED

Stainless steel is one of the most used construction materials in vacuum technology. Especially in XHV applications a high temperature treatment (vacuum firing) is commonly used to reduce outgassing of this material. There is a considerable body of work on outgassing of hydrogen from stainless steel. The results are basically described by two models: the diffusion limited model and the recombination limited model. Since recombination is strongly related to surface morphology and composition, surface characterization has been performed by atomic force microscopy (AFM) and scanning tunneling microscopy (STM). The surface near composition has been measured by atom probe depth profiling analysis. After vacuum firing a significant change in surface morphology can be observed in AFM and STM. The high temperature treatment leads to a complete reconstruction of the surface. The recrystallization process leads to an increase of the overall surface roughness with deep grooves up to 1000 nm in depth at the grain boundaries. On top of the crystallites wide flat terraces over 100 nm in width bounded by bunched atomic steps and facets can be observed. The high resolution STM micrographs additionally show stacking faults and local defects on these terraces assigned to (111) planes. The atom probe depth profiling analysis on vacuum fired samples results in a noticeable surface enrichment of nickel and certain depletion of chromium in the first atomic layer. In the second atomic layer chromium enrichment was measured. From the knowledge of surface structure and surface composition a recombination limited outgassing is very unlikely. Comparison with experimental studies on hydrogen desorption by thermal desorption spectroscopy strongly support the explanation by the diffusion limited model. It can be assumed that subsurface defects form traps with different energetic levels. The increase in diffusion energy after emptying the higher subsurface levels may also explain the observed outgassing behaviour of stainless steel.

\*Work supported by province of Styria, Austria Zukunftsfonds project P119.

2:40pm VT1-ThA3 Monte Carlo Simulation of Temperature Programmed Desorption Including Binding Energies and Frequency Factors Derived by DFT Calculations, P. Thissen, O. Ozcan, Max-Planck Institut for Iron Research, Germany, D. Diesing, Institut of Physical Chemistry Essen, Germany, G. Grundmeier, Institut of Macromoleculare Chemistry Paderborn, Germany

Temperature-programmed desorption (TPD) techniques are important methods for the determination of kinetic and thermodynamic parameters of desorption processes or decomposition reactions. A sample is heated with a temperature program  $\beta(t) = dT/dt$  (with the temperature T usually being a linear function of the time t) and the partial pressures of atoms and molecules evolving from the sample are measured, e.g. by mass spectrometry. When experiments are performed using well-defined surfaces of single-crystalline samples in a continuously pumped ultra-high vacuum (UHV) chamber then this experimental technique is often also referred to as thermal desorption spectroscopy (TDS). A Monte Carlo model has been developed for describing the temperature-programmed desorption of adsorbates from single crystal surfaces. The developed Monte Carlo Program requires the input of frequency factors and unity bond order binding energies BE (for the top position) for every bond under examination. For the first time the required values are now calculated using a DFT code. The virtue of both methods (Monte Carlo and DFT) is combined in the present work. Our new model takes into account the effects of surface diffusion, the influence of surface-adsorbate (S-A) and adsorbateadsorbate (A-A) interactions and the coverage dependence of the activation energy for desorption derived by precise calculations on an atomically defined level. The inclusion of localized (S-A) and (A-A) interactions has a pronounced effect on the shape of the predicted TPD spectrum. Only a single peak is observed in the absence of (S-A) and (A-A) interactions, whereas multiple peaks are found when these interactions are included. The inclusion of (S-A) and (A-A) interactions is also shown to produce a

nonlinear decline in the activation energy for desorption as a function of increasing adsorbate coverage.

3:00pm VT1-ThA4 Temperature Programmed Desorption Measurements of the Binding Energy of Water to Stainless Steel Surfaces, J.H. Hendricks, P.J. Abbott, National Institute of Standards and Technology, P. Mohan, NPL India, J.P. Looney, Brookhaven National Laboratory

The presence of water vapor is the limiting factor in achieving ultra-high vacuum (UHV) in an unbaked stainless steel system. While the "water problem" has been of scientific and technical interest for many decades, fundamental measurements of water interactions with stainless steel systems are not well characterized, including the binding energy and sticking coefficient of water on stainless steel. In addition, outgassing rate measurements of water from stainless steel surfaces are typically hampered by the problem of re-adsorption, leading to inaccurate measurement results. The NIST Pressure and Vacuum group has undertaken a study of the binding energy of water to stainless steel surfaces. A temperature programmed thermal desorption apparatus was constructed for this purpose and will be described in detail. The apparatus uses computer control to linearly heat a stainless steel filament at a rate of 3 °C/s while a quadrupole mass spectrometer detects the thermally desorbed species. The system is designed with a high pumping speed to minimize the problem of readsorption/desorption, and has reproducibly shown a water thermal desorption becorption, desorption, first employed by Redhead in 1962<sup>1</sup> was used to determine the activation energy, or binding energy of water to stainless steel. This technique has reproducibly yielded a water binding energy between 25.6 and 26.0 kcal/mole. These results may justify lower baking temperatures than are traditionally used for achieving UHV. Future work will focus on the apparent interplay of water desorption and hydrogen desorption observed during UHV system bakeouts

<sup>1</sup> Redhead, P.A., Vacuum, 12, 203 (1962).

### Vacuum Technology

Room: 618a - Session VT2-ThA

### Large Vacuum Systems

Moderator: N. Peacock, MKS Instruments, Inc.

3:40pm VT2-ThA6 Recent Advances to Enhance Space Simulation, F.G. Collins, The University of Tennessee Space Institute INVITED Accurate ground-based simulation of low earth orbit (LEO) conditions experienced by a satellite has proven to be a challenge. The continuous progress that has been made toward this goal will be reviewed. A satellite in LEO has a speed relative to the atmosphere of approximately 8 km/s. The neutral atmospheric molecules exchange momentum upon collision with the surfaces of the satellite, leading to drag, lift, and moments, but ground facilities still have trouble simulating pure beams of this speed for the relevant atmospheric gases in their ground state. A facility that is making progress toward this goal will be described. The most important atmospheric molecule, atomic oxygen, collides with ram-direction satellite surfaces with a relative energy of 5 eV. Energetic atomic oxygen atoms plus solar UV radiation produce synergistic effects that result in many chemical reactions on or in the vicinity of the outer satellite surfaces. These can lead to structural or operational damage and the spacecraft glow phenomena. It is desirable to generate large beams of atomic oxygen in the ground state, with the atoms possessing energy of 5 eV. Several techniques for attempting this will be reviewed. Solar radiation has a wide spectrum. The UV spectrum is a composite of many emission lines and continuum, which must be simulated using special lamp systems. Satellite surfaces are exposed to high energy protons, electrons, and other particles. These are simulated in combined effects space simulation chambers for materials degradation studies. Thruster plumes, surface outgassing, and liquid dumps lead to surface contamination. Contamination can reduce the effectiveness of thermal control paints, the output of solar cells, and the effectiveness of optical lenses. Some electric thrusters exit directly to the vacuum of space, which must be simulated if the thruster plume is to be accurately simulated. Specially designed cryogenic pumps designed to simulate the conditions that these electric thrusters will experience in orbit will be described. All of the facilities to be reviewed require special diagnostic instrumentation, much of a specialized type. Some of this instrumentation will be described

and the limitations of older techniques will be noted. Satellites in LEO also are immersed in a plasma but the effects of plasma charging, which has been well reviewed elsewhere, will not be covered. Links to inventories of space simulation chambers will be given.

4:20pm VT2-ThA8 Performance of a Unique Cryogenic Pumping System for Spacecraft-Thruster Interaction Studies, *C.G. Ngalande*, University of Southern California, *A.D. Ketsdever*, Air Force Research Laboratory, Edwards AFB, *S.F. Gimelshein*, University of Southern California

With the advent of advanced propulsion systems, the interactions of spacecraft thruster plumes and spacecraft materials is receiving renewed attention. Chamber IV of the Collaborative High Altitude Flow Facility (CHAFF-IV) was designed to obtain high fidelity spacecraft-thruster interaction data. CHAFF-IV uses a total chamber pumping concept by lining the entire interior of the chamber with an array of cryogenically cooled surfaces. The main pumping surface consists of a unique radial fin array which allows for the pumping of both neutral and ion effluents. A Monte Carlo numerical simulation has been performed to investigate the pumping efficiency of the radial fin array. In general, it has been found that longer fin widths and smaller fin thicknesses result in higher pumping efficiency. For a particular geometry, there is an optimum fin-separation distance at which the radial fin array pumping efficiency is maximum. A comparison of the pumping efficiency of the radial fin array with a flat pumping surface has shown that particles with high sticking coefficient such as neutrals will be pumped better with flat panel whereas particles with low sticking coefficients such as ions will be efficiently pumped with the radial fin array. CHAFF-IV is expected to pump, not only plume, but also sputtered material. Since ions are highly energetic, they will cause sputtering of both the array material and the pumped molecules. If not properly accounted for, these two populations can substantially increase the overall magnitude of pressure in the chamber making highly accurate tests impossible<sup>T</sup>. The Monte Carlo simulation has also been used to investigate CHAFF-IV's ability to pump these sputtered particles. A set of experiments has been performed to investigate the pumping efficiency of the radial fin array as manufactured. These experiments compared the radial fin results to a more traditional flat plate pumping surface with a neutral plume. These results indicate that there are flow regimes in which the radial fins are more efficient at pumping neutral molecules than a flat surface.

<sup>1</sup> Ketsdever, A.D., "Design Considerations for Cryogenic Pumping Arrays in Spacecraft-Thruster Interaction Facility", Journal of Spacecraft and Rockets, Vol 30, number 3, 400-410, 2001.

### 4:40pm VT2-ThA9 Outstanding Problems in Vacuum Gas Dynamics from an Industrial Point of View, *M. Wüest*, INFICON Ltd, Balzers, Liechtenstein

Many industrial vacuum processes in the semiconductor, coating, tribology, lighting or food packaging industry occur in the transitional flow regime in the pressure range between  $10^{-3}$  - 1 mbar. Industry wants high throughput in its vacuum processes, which requires fast pumping and venting. To achieve an optimum equipment design, the conductance of the vacuum flow path needs to be calculated. This is not an easy endeavour, as many assumptions in the derivation of the analytical conductance formulas are violated in the complicated non-symmetrical process equipment geometries and nonstationary process conditions. Modelling can also become quite difficult, especially if many different flow regimes need to be considered. Water outgassing is a critical process as it heavily influences the pumpdown time. However, our present understanding of the process is incomplete. There are two physically different models, namely the isothermal reversible adsorption and the diffusion-controlled outgassing models, to explain water outgassing. We also do not know the sticking coefficient of water on stainless steel very well. In this talk I will highlight a few outstanding vacuum gas dynamics problems from an industry perspective.

5:00pm VT2-ThA10 Minimizing Contamination to Multilayer-Dielectric-Diffraction Gratings within a Large Vacuum System, B. Ashe, K.L. Marshall, D. Mastrosimone, C. McAtee, University of Rochester The University of Rochester's Laboratory for Laser Energetics is in the final stages of constructing the OMEGA EP short-pulse, petawatt laser system. A critical component for OMEGA EP is the grating compressor chamber (GCC). This large (12,375-ft<sup>3</sup>) vacuum chamber contains critical optics where laser-pulse compression is performed at the output of the system on two 40-cm-square-aperture, IR (1054-nm) laser beams. Critical to this compression, within the GCC, are four sets of tiled multilayer-dielectric (MLD) diffraction gratings that provide the capability for producing 2.6-kJ output IR energy per beam at 10 ps. The primary requirements for these large-aperture (43-cm x 47-cm) gratings are high diffraction efficiency greater than 95%, peak-to-valley wavefront quality of less than @lamda@/10 waves, and high laser-induced-damage threshold greater than 2.7 J/cm<sup>2</sup> at 10-ps measured beam normal. Degradation of the grating laserdamage threshold due to adsorption of contaminants must be prevented to

maintain system performance. The presence of extrinsic contaminants (either particulate or molecular) in the vacuum system puts the MLD gratings at risk with respect to lowered damage threshold. A number of protocols have been developed and implemented at LLE to minimize MLD grating contamination and characterize the performance of the GCC vacuum chamber. In this paper, we describe the GCC vacuum chamber and component cleaning procedures, the qualification, testing methods, and studies undertaken for materials intended for use within the chamber, the use of absorptive getters to protect the gratings from molecular contamination, and the protocols necessary for the integration and operation of the MLD gratings. This work was supported by the U.S. Department of Energy Office of Inertial Confinement Fusion under Cooperative Agreement No. DE-FC52-92SF19460, the University of Rochester, and the New York State Energy Research and Development Authority. The support of DOE does not constitute an endorsement by DOE of the views expressed in this article. Key words: laser-pulse compression, vacuum chamber, cleaning, particulate contamination, molecular contamination.

## **Thursday Afternoon Poster Sessions**

Vacuum Technology

Room: 4C - Session VT-ThP

### Vacuum Technology Poster Session (including Student Poster Competition with Cash Award)

#### VT-ThP1 Design and Construction of a Vacuum Tube Furnace with High Voltage Field for the Growth of Silicon Nanowires, C.A. Adams, J.J. Register, University of South Florida

Construction of a Vacuum Tube Furnace with High Voltage Field for the Growth of Silicon Nanowires. The goal of this project is to build a vacuum tube furnace providing up to 1150 degrees Celsius in a vacuum of approximately 30 mTorr and an electric field of 5V/µm in density to the sample. Feed and purge gasses will need to be fed through into the chamber. The tube furnace will be constructed of a ceramic cylindrical heater element mounted in a stainless steal housing with carbon board insulation surrounded. The tube will be Quartz approximately 12.5 cm diameter and 122 cm long and 2.4 cm thick with an open end and the other end reducing to a 3/8 inch tube. A stainless steal termination will be designed and built to provide a vacuum seal to the open end of the tube. This termination will use viton gasket compression seal and end in a vacuum door. All feedthroughs will take place through the side walls of the termination collar. The vacuum system will consist of a two stage mechanical pump with a foreline trap and a thermocouple vacuum gage. MKS vacuum gage and mass-flow controllers will be integrated with Labview for operational programming and data recording. Labview will also be used to record the high voltage run time and values, control and record the temperature. The furnace heater element will be powered by an Omega PID controller coupled with a SSR.

#### **VT-ThP2** Experimental Measurements of Thermal Accommodation Coefficients for Microscale Gas-Phase Heat Transfer, W.M. Trott, D.J. Rader, J.N. Castañeda, J.R. Torczynski, M.A. Gallis, Sandia National Laboratories, *L.A. Gochberg*, Novellus Systems, Inc.

An experimental apparatus is described that measures gas-surface thermal accommodation coefficients from the pressure dependence of the conductive heat flux between parallel plates separated by a gas-filled gap. Heat flux between the plates is inferred from measurements of temperature drop between the plate surface and an adjacent temperature-controlled water bath. Thermal accommodation coefficients are determined from the pressure dependence of the heat flux at a fixed plate separation. The apparatus is designed to conduct tests with a variety of gases in contact with interchangeable, well-characterized surfaces of various materials (e.g., metals, ceramics, semiconductors) with various surface finishes (e.g., smooth, rough). Experiments are reported for three gases (argon, nitrogen, and helium) in contact with pairs of 304 stainless steel plates prepared with one of two finishes: lathe-machined or mirror-polished. For argon and nitrogen, the measured accommodation coefficients for machined and polished plates are near unity and independent of finish to within experimental uncertainty. For helium, the accommodation coefficients are much lower and show a slight variation with surface roughness. Two different methods are used to determine the accommodation coefficient from experimental data: the Sherman-Lees formula and the GTR formula. These approaches yield values of 0.87 and 0.94 for argon, 0.80 and 0.86 for nitrogen, 0.36 and 0.38 for helium with the machined finish, and 0.40 and 0.42 for helium with the polished finish, respectively, with an uncertainty of ±0.02. The GTR values for argon and nitrogen are generally in better agreement with the results of other investigators than the Sherman-Lees values are, and both helium results are in reasonable agreement with values in the literature.

### VT-ThP3 Sputter Deposition System for High Throughput Fabrication of Composition Spreads, J.M. Gregoire, R.B. van Dover, J. Jin, F.J. DiSalvo, H.D. Abruna, Cornell University

We describe a custom built sputtering system that can deposit composition spreads in an effectively UHV environment but which does not require the high-throughput paradigm to be compromised by a long pumpdown each time a target is changed. The system employs four magnetron sputter guns in a cryoshroud (getter sputtering) which allows elements such as Ti and Zr to be deposited with minimal contamination by oxygen or other reactive background gasses. The system also relies on custom substrate heaters to give rapid heating and cooldown. The effectiveness of the gettering technique is evaluated, and example results obtained for catalytic activity of a pseudoternary composition spread are presented.

# VT-ThP4 How Does One Turn a Research-Based Molecular Beam Epitaxy System into a Reliable Training Tool?, M.-R. Padmore, E.I. Altman, V.E. Henrich, F. Walker, Yale University

Electronic devices are simultaneously decreasing in size while increasing in their importance to our everyday lives. Thin film crystalline growth is necessary for the production and integration of micro- and nanoelectronics. The Molecular Beam Epitaxy (MBE) system is one of the most widely used methods of achieving this growth. As such, it is valuable to train students in the use and applications of MBE as early as at the undergraduate level. However, in using the system to train less-experienced users, the chance of system failure increases dramatically. Thus, a system must be built which can, through the use of preventative interlocks and user-friendly interfaces, easily and cost-effectively be incorporated into an instructional setting. A major problem with many preventative measures is that they are implemented using administrative and/or procedural controls. These types of controls block the transparency of the process by not allowing the students to interact with the system itself. This system is designed to be "hands-on", allowing the trainees to see the basic science behind the technology they are using, without reducing the reliability of the system. By using engineering controls such as automatic valves and computerized shutdown interlocks, the main failures which arise from: a.) the sample transfer mechanism; b.) the ultra-high vacuum requirements; and c.) the water cooling requirements; can be avoided without severely decreasing the transparency of the system's process and with an increase in ease of use. An evaluation of the transparency, user-friendliness, and reliability of the system were conducted by allowing a small group of students of various backgrounds to perform experiments mapping out surface phase diagrams on silicon surfaces. Preliminary results of this study show that an effective set of engineering controls can be designed. The conflicting problems between transparency and reliability addressed in designing this system are not specific to Molecular Beam Epitaxy, but to any educational institution whose mission is to create reliable training tools in all fields of engineering and technology.

### VT-ThP5 Vacuum Chamber Design at National Synchrotron Light Source II, J.-P. Hu, H.-C. Hseuh, C. Foerster, Brookhaven National Laboratory

National Synchrotron Light Source II (NSLS-II), proposed to be built at Brookhaven National Laboratory, will be a 3-GeV 800-meter circumference 3rd-generation synchrotron radiation facility. To provide a highly-stable and highly-focused synchrotron beam for advanced research, vacuum pressure at 10-9 Torr or below in the storage ring during normal operation is deemed critical. The approach for achieving such ultra-high vacuum would rely on the effective arrangement of non-evaporable getter strips, titanium sublimation pumps, and sputter ion pumps along the ring chamber, under proper in-situ baking and beam conditioning. The ring chamber will be made of aluminum through extrusion followed by machining, from which side-ports and ante-chamber can be precisely made to accommodate photon absorbers, lumped and distributed pumps. To test the design of the vacuum system, Monte Carlo-based Molflow and gas diffusion solver Vaccalc codes are utilized to calculate the pressure variation in channels hosting the electron beam, based on different pumping setup in models. Spare aluminum chambers of the Advanced Photon Source in Argonne Laboratory, featuring similar cross section as the NSLS-II proposed, will be used in bench test of pumping performance, thereby verifying pressure profile from code simulation. Updated design to improve ring vacuum and thus beam quality will be presented per project progress. (Work performed under auspices of the United States Department of Energy, under contract DE-AC02-98CH10886).

### VT-ThP6 Viscosity and Diffusion Coefficient for a Gas Mixture Flow in a Tube, Valid Over the Whole Range of Knudsen Numbers, *M. Vukovic*, Tokyo Electron U.S. Holdings Inc.

The viscosity and diffusion coefficients for a two-gas mixture, valid over the whole range of Knudsen numbers (Kn), are obtained by applying the generalization procedure of Beskok and Karniadakis (Microscale Thermophysical Engineering 3, 43, 1999) to the Boltzmann equation solutions of a gas mixture flow in a pipe by Sharipov and Kalempa (J. Vac. Sci. Technol. A 20(3), 814.,2002). The transport coefficients are expressed in terms their continuum limit value, multiplied by a correction factor that depends on Kn and the species masses and relative concentration. These coefficients are applied to the problem of gas counterflow in the Kn=1 range. VT-ThP7 New Apparatus for Testing Hermetically Sealed Packages of Electronic Devices, *M. Kinugawa*, *H. Kurokawa*, *S. Takagi*, Mitsubishi Electric Corp., Japan, *H. Kawata*, Wave Technology Inc., Japan

Hermetically sealed packages are widely applied to optical and highfrequency devices to maintain high reliability. The inert gasses, which are usually inserted in such packages, might contain such impurities as moisture that could damage the device. Therefore, it is crucial to know the variety and the amount of impurities in the filled gas. In this study, we show a new technique for analyzing gas in sealed packages. The advantage of our new technique is that analysis precision does not depend on package size. The testing apparatus consists of a sample chamber and an analysis chamber; they are both connected to a vacuum-tight valve and an exchangeable orifice. The analysis chamber has a quadrupole mass spectrometer and is exhausted continuously by a turbomolecular pump. The sample chamber has a rotating vacuum feedthrough that can mount several types of sample stages, a perforator, a viewport, and another pumping system. After setting sample packages on the sample stage, the sample chamber is pumped down by the pumping system. Then the pump is switched off, and the valve between the two chambers is opened, so the sample chamber will only be exhausted from the analysis chamber through the orifice. A pinhole is made on the sample package by the perforator. The gas in the sample package comes out and flows through the orifice from the sample chamber to the analysis chamber, and then the mass spectrometer in the analysis chamber detects the gas. We can get the gas composition from the integral ion intensity measured by the mass spectrometer and its ion sensitivity. The conductance of the orifice is determined by considering the sample size and the maximum vacuum pressure in the analysis chamber to keep the pressure a little lower than the working upper limit of the mass spectrometer. This technique allows accurate measurements of gas composition regardless of size package. We have already applied this technique to the development of new devices with higher reliability.

### VT-ThP8 Optimization of a Multitarget Sputtering System for the Production of Magnetic Tunneling Junctions and Multilayers, A. Chiolerio, P. Martino, Politecnico di Torino, Italy

Our work consists in a renovation project for an obsolete multitarget sputtering system, previously used for industrial purposes, in order to revert it to research objectives with a cost-effective operation, saving as many original components as possible. The system was equipped with a control rack, a large cylindrical vacuum chamber and a complete rotary turbomolecular pump evacuation subsystem; substrates where inserted into a slit close to a vacuum oven by vertically lifting the whole chamber above the steel basement by means of an oleodynamic piston. The rotating oven slit was manually positioned above one of the three target sources allowing a rotation of 270°. Our newly realized system maintains the original control rack, the evacuation subsystem and the basement. A smaller cylindrical chamber has been designed in order to reduce both void spaces and evacuation time; all inner parts of the AISI 316L walls have been polished to minimize the outgassing rate while the opening is assisted by a vertical lift motor and performed only for scheduled maintenance, because a loadlock chamber is now connected to the operation chamber via a gate valve. This smaller buffer chamber has been designed either to insert and recover substrates or to serve as a second process chamber for the realization of tunneling barrier thin oxyde layers, where an oxygen line inlets the reactive gas and a sapphire window allows UV radiation to assist the critical step. Specimens are transferred to the carrying slit inside the process chamber via a magnetic manipulator; this slit is positioned above the desired sputtering source by means of an AC brushless motor operated by a remote control software. The rotating subsystem allows a full rotation (360°), the running cables being substituted by an ad hoc designed electrical rotating contact that ensures quite low electric field at contact point, no debris and appropriate friction during operation. A particular manual implementation allows one to choose between the simultaneous rotation of two coaxial axes and the movement of only one of them. The first axis is connected to the substrate slit and the other one to the shutter, which may interrupt the deposition process or interpose suitable masks to transfer geometries to the substrates. This solution ensures a higher vacuum level, with only one mechanical feedthrough, as well as much lower costs.

### VT-ThP9 Influence of the Adsorption Gas on Friction Coefficient and Wear Track., A. Kasahara, M. Goto, Y. Pihosh, M. Tosa, NIMS, Japan

Surface modification of sliding motion materials is inevitable to reduce friction as well as outgassing in a vacuum. We have therefore studied the development of advanced vacuum motion materials by control of surface roughness on a submicron scale. We have successfully found that any material sheet with about 100nm-250nm surface roughness showed same friction coefficient in a vacuum as at an atmospheric pressure for Type 304 austenitic stainless steel materials and anodic oxidation processed aluminum materials. Materials with except 100nm-250nm surface roughness shows a different friction coefficient for the friction measurement in a vacuum and at an atmospheric pressure. Type 304 austenitic stainless steel sheets had a larger friction coefficient in a vacuum than at an atmospheric pressure. The anodic oxidation processed aluminum sheets had a smaller friction coefficient in a vacuum than at an atmospheric pressure. We therefore studied the relation between friction coefficient and cross-section shape of wear track depth by an atomic force microscope (AFM) Type 304 austenitic stainless steel sheets had a deeper wear track in a vacuum than at an atmospheric pressure probably because the increase in friction coefficient to the desorption of the adsorption gas as a lubricant. The anodic oxidation processed aluminum sheets had shallow wear track in a vacuum than at an atmospheric pressure probably because the adsorption force generated by the applied load was reduced by the desorption of the adsorption gas. As a result, it was thought that the absorption layer on the materials surface influenced by the surface roughness had an important role on friction.

#### VT-ThP10 Atomic Layer Deposition Reactor with In Situ Diagnostics for Studying Gas-Surface Interactions, V. Rai, B.N. Jariwala, S. Agarwal, Colorado School of Mines

In this presentation, the authors will describe the design and fabrication of a custom vacuum chamber for studying the heterogeneous surface chemistry during thin film deposition. The chamber is ideally suited for investigating the film growth mechanism during atomic layer deposition (ALD). The reactor consists of a cylindrical stainless-steel vessel, which is 10 inches in diameter, 6 inches in height. The reactor volume has been minimized to reduce the residence time of the gases to minimize the purge duration in an ALD cycle. The chamber is equipped with multiple ports for instrumentation, sample manipulation, and in situ surface and gas-phase diagnostics. The substrate is clamped to a heated plate, and the deposition temperature can be varied from 40 to 300 °C. The reactor is pumped by a 240 l/s turbomolecular pump, which provides a base pressure of  $9 \times 10^{-8}$ Torr. The chamber also has a parallel-plate, capacitively-coupled plasma source operating at a frequency of 13.56 MHz to generate radicals for plasma-assisted ALD. The distance between the plates can be varied from 4 cm to 9 cm using flexible linear motion bellows. The oxygen inlet into the reactor is equipped with an ozone generator to provide an alternate oxidant during metal oxide ALD. To deliver controlled amounts of low volatility precursors, we employ pressure-based mass flow controllers that require an inlet pressure of only 4 Torr and do not heat the temperature-sensitive precursors. The reactor is equipped with three in-situ diagnostic tools - (1) attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy, (2) quadrupole mass spectrometry (QMS), and (3) quartz crystal microbalance (QCM). In addition, a set of ports is available for in situ spectroscopic ellipsometry. The combination of ATR-FTIR spectroscopy and QCM provide sub-monolayer sensitivity to surface adsorbates. The QMS, which is placed in a differentially pumped housing, is used to detect the surface reaction products. Specifically, we will present results from gas-surface interaction studies during the ALD of titanium dioxide using metal precursors such as titanium tetrachloride and titanium isopropoxide, and oxidants such as water, ozone, and O radicals.

### VT-ThP11 A Real Time Monitoring Method on the Decomposition Degree of MOCVD Precursor through an Ultrasonic Diagnosis Method, J.Y. Yun, S.W. Kang, D.J. Seong, KRISS, S. Korea

This study proposes a method for monitoring the decomposition state of the metal-organic precursor which is used for the Metal Organic Chemical Vapor Deposition (MOCVD) system. As the precursor of MOCVD is highly likely to decompose due to the instability of its chemical structure during processing, critical problems are generated for thin film formation and its yield in this case. Although real time monitoring technology on the decomposition degree of these precursors is essential for the next generation semiconductor process, both commercialized technology and fundamental research have scarcely been accomplished. Therefore, this study endeavors for acknowledgement by the semiconductor industry with a proposal for a real time monitoring method on the decomposition degree of the precursor through an ultrasonic diagnosis method.

# **VT-ThP12** Development of a Static Expansion Vacuum Standard at the National Institute of Standards and Technology, *J.H. Chow*, *P.J. Abbott*, National Institute of Standards and Technology

The NIST Pressure and Vacuum Group maintains sub-atmospheric pressure standards that range from  $10^{-7}$  to  $10^{+5}$  Pa. These consist of orifice flow standards from  $10^{-7}$  to 10 Pa and Ultrasonic Interferometric Manometers from  $10^{-1}$  to  $10^{+5}$  Pa. A new Static Expansion Standard is in development with an operating range of  $10^{-3}$  to  $10^{+3}$  Pa. The standard is composed of two spherical stainless-steel vessels with a volume ratio of about 100:1. The two vessels are interconnected with a stepper motor driven valve. An initial pressure of gas in the small volume is measured to high accuracy using a resonant silicon gauge and then is expanded into the evacuated larger volume. This expansion results in a pressure reduction of about 100. Further

evacuation and expansion cycles are used to produce lower pressures. The system is temperature controlled to within 0.1C with a thermoelectrically cooled enclosure. This new standard provides many useful benefits: First, the Static Expansion Standard generates pressures that encompass both the orifice flow and manometry standards without relying on gas flowmeters; Second, the standard enables fast and simple calibration of spinning rotor (SRG's) and capacitance diaphragm gauges (CDG's). Currently these calibrations require a very skilled technician and are done at very high cost to customers; Third, the system allows for a convenient comparison of manometer and vacuum standards using CDG's and SRG's respectively. Work is ongoing to characterize the system, including determining the volume ratio and estimating the uncertainties.

#### VT-ThP13 Custom-Designed Very-High Vacuum Chamber for Growth of Large Area Silicon Nanowhiskers Arrays via an Ion-Enhanced Vapor-Liquid-Solid (VLS) Mechanism, M. Bettge, University of Illinois at Urbana-Champaign, D. Abraham, Argonne National Laboratory, S. Burdin, S. MacLaren, I. Petrov, E. Sammann, University of Illinois at Urbana-Champaign

A VHV chamber was custom-designed for experimental growth of silicon nanowhisker arrays via a novel ion-enhanced Vapor-Liquid-Solid (VLS) technique. Growth was to be carried out on a self-organized metal seed layer on a Si or SiO<sub>2</sub> surface. A reactive magnetron sputtering system was needed to supply atomic Si to the growth surface under concurrent highenergy ion irradiation. The successful implementation of this growth technique required sample temperature control to 450°C, a bias voltage up to 3kV, and a reactive plasma environment. Additional deposition capabilities and process controls for several inert and reactive gases were also required. The design goal was to develop an economical system using standard vacuum hardware that allowed handling of two-inch Si wafers. A wafer stage using a halogen reflector bulb for temperature control was designed to meet the requirements for whisker growth. Protection of the insulators and wafer handling proved to be especially challenging during the design of this stage. The chamber design also included capabilities for wafer storage and wafer transfer between two DC magnetron sputtering stations and a miniature evaporator custom-designed to fit a 2<sup>3</sup>/<sub>4</sub>-flange. This presentation will describe the realization of the chamber design, which made possible the processing of nearly four hundred samples to date. Ultimately, this enabled the growth of aligned Si nanowhisker arrays at temperatures below 200 °C and at rates up to 200 nm/min. Growth can take place on any substrate on which a thin Si film can be deposited.

### VT-ThP14 A Low Pressure Chemical Vapor Deposition (LPCVD) System Designed for Epitaxial Growth of 3C-SiC on Si, *M.P. Orthner*, *F. Solzbacher, L.W. Rieth, E. Jung*, University of Utah

Silicon carbide (SiC) can be used as electronic material at high temperatures (>500°C) and in aggressive/corrosive gas and fluid media. Depositing thin films of 3C-SiC on Si will permit use of existing Si processing technologies on the substrate while the 3C-SiC is processed as the active layer. Cubic silicon carbide (3C) is the only polytype that can be epitaxially grown on Si. This material combination makes SiC useful in pressure sensors, accelerometers, and for encapsulation. A key problem for 3C-SiC is the number of defects formed during growth due to a 20 % lattice mismatch. The deposition of 3C-SiC on Si by low pressure chemical vapor deposition (LPCVD) has been reported using a number of different precursors and a wide range of operating conditions. Growth temperatures range from 650°C to 1400°C and pressures range from atmospheric to mTorr. An LPCVD reactor with unique hot zone geometry has been developed to achieve the required growth conditions to study the growth of 3C-SiC on Si. The system is a cold wall design using a 304 stainless steel (SS) water cooled vacuum chamber. Pressure feedback is controlled using a butterfly valve and MKS capacitance manometer down to 50 mTorr and is currently limited by the Alcatel 813B dry pump. The resistive heater is a 7" diameter graphite spiral used to heat 2", 3", 4", and 6" Si substrates. The heater is mounted 1" above the graphite wafer chuck that is rotated by a stepper motor to increase uniformity. An Omega IR2P optical pyrometer and temperature controller are used in conjunction with the 180A phase angle fired SCR to measure and control the substrate temperature. Substrate temperatures in excess of 1400°C have been achieved. A custom designed horizontal flow showerhead manufactured from 316L SS is located on the side of the wafer chuck. The gases pass horizontally over the heated Si substrates to form thin films. Silane and propane precursors are used to grow the 3C-SiC using the conventional carbonization growth technique first introduced by S. Nishino. Initial deposition of very thin (< 100nm) 3C-SiC on Si (100) substrates have been grown in the described reactor. Deposition conditions maintained a process pressure of 250 mTorr with temperatures ranging from 1200°C to 1400°C. The chemical composition, crystal morphology and surface properties were investigated in relation to the growth temperature.

VT-ThP15 Method of Diagnosing Mechanical Endurance of Dry Vacuum Pumps to High Throughput Environment, J.Y. Lim, W.S. Cheung, Korea Research Institute of Standards and Science, B.H. Moon, Samsung Electronics Company, Korea, Y.H. Shin, Korea Research Institute of Standards and Science

Dry Vacuum Pumps are encountering rapid expansion of harsh and high throughput applications to semi-conductor/display industry. Besides their ability of making superior clean environment, they are normally exposed to clean to very harsh gas applications, thus often very easily contaminated with process byproducts in the forms of hard particles or coatings on the overall interior of the pumps. For this reason, even before installation in the process lines, detection of latent endurance for the pumps is a very important factor to diagnose primary mechanical, functional ability and internal controllable parameter settings during actual processes. With 40 to 300 slm of continuous dry air flow for one to three hours dependent on the pumping speeds, ten dry vacuum pumps carefully selected from six worldwide manufacturers were differently responded due to their internal structures, cooling systems, and breaker settings for body protection due to high temperature. Parameters such as inlet pressure, power consumption, and sound/vibration as well as parameters from SPM (single pump monitoring system) were carefully analyzed to diagnose the latent endurance of each pump. Characteristics mappings for all parameters were established for 1200 and 1800 m3/h dry vacuum pumps. In the case of total power consumption, 4 to 8 kW are manifested in range of atm. to 10-3 mbar. Dry pump body temperatures were very fluctuant in several pumps, thus minor or major modifications for their cooling systems may be necessary for those pumps. At the end of each test sequence, sudden vent test was performed to verify the pumping down characteristics as well as power consumption. Exposed to sudden avalanching mass flow, several pumps are diagnosed as relatively abnormal compared to their initial pumping down characteristics. Based on experimental results with integrated laboratory experimental system, we propose recommended characteristics design guidelines for dry vacuum pumps in the semiconductor and display industry in the course of serial evaluation periods.

# VT-ThP17 ISAC / SEBT Vacuum System at TRIUMF, I. Sekachev, D. Yosifov, TRIUMF, Canada

SEBT is the "Super" Energy Beam Transport line connecting the mediumbeta superconducting linear accelerator with the experimental hall in the ISAC-II building at TRIUMF. The SEBT vacuum system consists of five independent sub-sections comprised of nine 280 L/s (N) turbo-molecular, 5 dry mechanical pumps, 22 vacuum gauges, and 37 valves. A liquid nitrogen cooled trap and fast valve protect the Linac from pump oil migration and particular matter potentially originating from vacuum disruptions in the Experimental hall equipment. A control system with appropriate interlocks is used for system operation and protection in accordance to protocol.

### VT-ThP18 In-situ Diagnosis using FT-IR Spectroscope System Installed at the Exhaust Line of the Chamber and the Characterization of Al Metal Films with a New Precursor, S.W. Kang, J.Y. Yun, D.J. Seong, Y.H. Shin, Korea Research Institute of Standards and Science, I.D. Yang, J.Y. Shim, J.C. Oh, Quleap, S. Korea

In-situ Fourier transform infrared (FT-IR) spectroscope system was installed at the exhaust line of the chamber to monitor the by-products generated by gas phase reaction and surface reaction. The by-products of Al metal precursor (a new one) were changed as a function of deposition temperature and pressure and so on (deposition condition). The intensity of spectra measured at the exhaust line was also varied as a measured condition. In in-situ FT-IR spectroscopy studies vibrational spectroscopy reveals the gain and loss of the by-products as a function of the deposition condition. The behavior of the functional group (such as N-C, C-C, C-H, etc.) was monitored and from that, the temperature dependence of the film properties (ex: the film composition) could be explained. The basic properties of a new Al precursor will be introduced.

### VT-ThP19 Consideration for the Role of Surface Boundary Layer and the Mechanism of Hydrogen Desorption in Stainless Steel, K. Akaishi, University of Toyama, Japan

Tritium loaded stainless steel specimens of 0.5 mm thickness plate were prepared. When the specimen was chemically etched by about 70  $\mu$ m in thickness,the amount of tritium trapped within the top surface was measured by  $\beta$ -ray induced X-ray spectroscopy technique. When the chemically etched specimen was immersed in argon gas flow in atmosphere at ambient temperature, more than 99% of tritium released from the specimen was tritiated water, HTO, and the amount of released tritium was measured as a function of time by counting technique. In this paper to evaluate quantitatively the above experimental results, a model for hydrogen transport is proposed, and results of numerical simulation for the tritium release are shown. It is demonstrated that the numerical simulation well

predicts the amount of tritium released from the stainless steel specimen in the experiment. This work will be discussed as an issue of outgassing reduction in stainless steel materials.

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