## Vacuum Technology
## Room: C1 - Session VT-MoM

## Vacuum Contamination and Pumping
**Moderator:** M. Wüest, INFICON Ltd., Liechtenstein

8:20am **VT-MoM1 Silicon-based Surface Treatments for Improved Vacuum System Throughput, Inertness, and Corrosion Resistance**, ***D.A. Smith***, SilcoTek Corporation, *B.R.F. Kendall*, Elvac Associates **INVITED**

Tests of stainless steel components with a silicon-based deposition have shown significantly lower outgassing rates when compared with stainless steel components without a surface deposition. A variety of experiments illustrate the beneficial aspects of a silicon-based deposition in process vacuum systems. For outgassing performance, experimentation was developed for comparing otherwise identical samples having various surface treatments and/or coating types. The samples are heated and cooled in turn while the outgassing rates are recorded at temperatures up to 250 degrees C. For inertness performance, chromatographic and gravimetric data will illustrate the lack of adsorptive and catalytic behavior of a substrate with a silicon-based deposition. These depositions can also serve as an anti-corrosive barrier for processes hindered by frequent maintenance after exposure to environments that are corrosive to base materials. ASTM corrosion testing methods will be presented and discussed to better understand the anti-corrosive nature of these deposited surfaces. Process vacuum systems requiring rapid pumpdown, minimal metallic substrate exposure and contamination, and/or reduced corrosive attack may benefit from the characteristics capable with silicon diffusion coatings.

9:00am **VT-MoM3 Modeling Decontamination of Vacuum Chambers by Downstream Plasma Cleaning**, ***C.G. Morgan***, *R. Vane*, XEI Scientific, Inc.

Downstream plasma cleaning is an effective means for removing carbon contamination from vacuum chambers. The downstream plasma cleaning device is mounted on an unoccupied port on a vacuum chamber. When in use, the device creates oxygen radicals using a small leak of oxygen containing gas and a low power (5-20 W) radio frequency (RF) plasma. The oxygen radicals then flow through the vacuum chamber, ashing hydrocarbons. The rate of carbon decontamination has been shown to be dependent on a number of factors: RF power level, chamber pressure and geometry, distance between radical source and contamination, speed of the pumping system, and type of oxygen containing gas used. Decontamination rates can be accurately estimated if chemical models of both the oxygen radical reactions within the chamber and on the contaminant surface can be developed. These models are validated by physical data from two experiments varying the parameters listed above.

Data is presented from two experiments with oxygen radicals. Both experiments use a quartz crystal microbalance (QCM). In the first, a silver-coated QCM is placed in the vacuum chamber and subjected to the plasma cleaning process. Oxygen radicals will incorporate themselves into the QCM and increase its mass. The flux of oxygen radicals impinging on the QCM surface can be calculated using the Deal-Grove model of surface oxidation. By locating the silver-coated QCM in different locations of a vacuum chamber, a map of oxygen radical concentrations as a function of distance from the plasma can be made. In the second, a gold-coated QCM is contaminated with hydrocarbons. Test contamination is achieved by heating a small amount of hydrocarbon in a vacuum chamber and allowing the evaporation to recondense on the gold-coated QCM. The cleaning process is then initiated and an experimental trace showing mass loss from the gold-coated QCM as a function of cleaning time is obtained.

A chemical box model which assumes that once the plasma is lit there is a steady-state oxygen radical concentration within each box can be compared to the data from the silver-coated QCM experiments. The chemistry within each box is obtained by using a standard database of gas phase reaction rates. The second model focuses on the gas surface chemistry of decontamination. The results of the second model are compared with the data from the mass loss traces of the gold-coated QCMs. The combination of both models will provide a means to estimate rates of downstream plasma cleaning for any contaminated vacuum system.

9:20am **VT-MoM4 Methods for Measuring Outgassing for Qualification of Materials, Components and Systems**, ***N.B. Koster***, *R. Koops, E. van Zeijl*, TNO Science and Industry, Netherlands

Presently cleanliness requirements for vacuum systems in use for Extreme Ultra Violet Lithography (EUVL) are beyond what with standard procedures can be achieved. Especially the constraint that the system cannot be baked after assembly, whilst cleanliness better than UHV is needed, requires special measures with respect to manufacturing and qualification. Because of the constraint of not being able to bake the system we refer to this type of vacuum as Ultra Clean Vacuum (UCV). This presentation will focus on outgassing measurement methods for qualification of materials and components. Traditionally only the total outgassing is measured and reported, as can be found in many vacuum handbooks. In the case of EUVL, or other systems with high energetic particles, we distinguish several species of interest, like water and hydrocarbons and provide numbers for outgassing of these species as measured with a RGA. These measurements enable engineers to calculate total pressure and cleanliness of a system under design. Results of these measurements will be shown including a way of representing the data in a clear format. We also will show results of a new method for measuring hydrocarbon outgassing with the aid of a RGA when the outgassing levels are at the lower detection limit of the RGA.

9:40am **VT-MoM5 Outgassing Characterization of Elastomeric Seals Used in Semiconductor Wafer Processing**, ***M. Heller***, *S. Sogo, J. Chen, J. Legare*, DuPont Performance Elastomers L.L.C.

Many integrated circuit manufacturing processes operate in high or ultra high vacuum (UHV) environments. It is important that vacuum levels are maintained within specified limits to insure optimum process efficiency. While specification of an appropriate size vacuum pump for the system can insure that overall vacuum levels are maintained, outgassing from sealing materials can interfere in the process by changing the composition and morphology of the deposited layer. For instance, outgassing contaminants absorbed by the exposed substrate during the initial steps of the deposition process can induce undesired interactions at the interface level and consequently affect the grown film as well as the overall process. Therefore, it is important to understand the outgassing characteristics of elastomeric seal materials in order to select the appropriate material for a given application.

A methodology has been developed using a residual gas analyzer to measure the outgassing properties of elastomers. Results indicate that outgassing typically takes place in two stages. With some minor exceptions, the first stage involves the evolution of atmospheric gasses and absorbed moisture (i.e. nitrogen, water, oxygen, and carbon dioxide). The second stage and possibly of greater interest involves the evolution of gasses related to the thermal stability and decomposition of the material in question.

This paper compares the outgassing characteristics of three different types of elastomeric seals (perfluoroelastomers, fluorelastomers and silicones) typically used in semiconductor wafer processing. Data on outgassing rate as a function of time and temperature, and classification of gas species evolved for products in each material class are presented. While perfluoroelastomers offer the lowest outgassing rate at elevated temperatures, there can be some performance variation within this material class. The relationship between outgassing performance and elastomer formulation will also be discussed.

10:00am **VT-MoM6 Permeation Through Elastomers: Comparison of Viton® and Chemraz® 653 O-rings under Controlled Compression and Temperature**, ***N.T. Peacock***, MKS Instruments, HPS Products

Many types of elastomers are available and used for demountable seals in vacuum service. One important consideration in the selection of the elastomer material is the permeation rate. The permeation rate for gasses like helium can differ by orders of magnitudes for different seal materials. In this study, the gas load through a single O-ring due to permeation was compared for Viton® E and for Chemraz®653. The procedure was to use a MSLD (mass spectrometer leak detector) with helium and log the leak signal at intervals often a few seconds apart as a controlled flow of helium was applied to the seal. This was done both at room temperature and at elevated temperatures up to 140o C. The test O-ring was located in a specially constructed fixture that allowed an O-ring to be compressed to five different values ranging from 15% to 27% compression. Leak signals due to permeation through the 2-227 sized O-ring (nominal 0.139 inch cross section) were found to vary by orders of magnitudes. For instance at 22% compression and room temperature, the peak leak signal from the Viton® seal was approximately 1x10-10 mbar-l/sec while for the Chemraz®653 seal it was approximately 2x10-8 mbar-l/sec. When the seals were at 140o C with the same compression, the leak signals became approximately 1x10-8 mbar-l/sec for the Viton® and approximately 8x10-7 mbar-l/sec for the Chemraz®653 seal.

Leak signals due to permeation of these magnitudes are very important when troubleshooting or qualifying equipment since leak rate specifications on equipment are often lower. The same set up was also used to help find ways to distinguish a response on the leak detector due to a leak from a signal due to permeation. Using the rapid data recording and graphical display of the leak signal, it was found that permeation responses had a characteristic shape. With a response due to permeation, there is a short time before the response starts, a ramp up time, and then a slow decay. By contrast, signals from known leaks were shown to have a very rapid response time, and a quick decay or 'clean up' when the probe gas was removed. Using a graphing display, and comparing the response to known examples, operators can distinguish the two situations.

10:40am **VT-MoM8 Controlled Formation of Condensed Frost Layers in Cryogenic High Vacuum Pumps**, ***S.E. Syssoev***, *A.J. Bartlett, M.J. Eacobacci*, Brooks Automation Inc.

Cryogenic high vacuum pumps are used on a wide variety of vacuum substrate processing equipment, space simulation systems, and analytical instruments. They produce high pumping speeds for all gases and work over a wide range of pressures. Pumping residual gases occurs by cooling them to the point that they condense on the appropriate cryogenic surface. Thus, the pumping speed of the cryopump can be converted into deposition rate of the pumped gases onto cold surfaces inside the pump. The thickness of the deposited layer is uneven due to geometry of the cryopanels inside the pump. The majority of the trapped gas forms thick and comparable stable amorphous structures, while a significantly smaller amount of the pumped gas is participating in low rate deposition on those zones inside the pump that are less exposed to the gas flow. This low rate deposition leads to formation of polycrystalline films with complicated crystallographic structure even for the simple binary gas mixture widely used in the most applications in semiconductor industry. As with any thin film, this type of polycrystalline frost can be subject to appreciable residual stress due to structural defects. The concentration of such defects depend on operating conditions such as pumped gas composition, pressure, rate of deposition, and condensation temperature. For the stressed film there is always a certain film thickness (critical thickness, [1]) after which the film can exhibit one of the possible cracking pattern – surface crack, channeling, or debond. Defects in the condensed solid gas films grown inside the cryopump can lead to spontaneous delamination resulting in frost flakes being ejected from the array surface with subsequent sublimation on the warmer surfaces of the pump. Sporadic sublimation of delaminated flakes lead to unwanted pressure variations, or bursts, inside the vacuum system. This report discusses the types of film formations found in a typical cryopumping array structure and summarizes the development of a new cryopump with increased capacity and elimination of sporadic pressure bursts occurring during the cryopumping of type II gases. The pump employs the GM refrigeration cycle and is a further modification of the Brooks Automation On-Board IS 8F cryopump [2]. The test results showing pressure bursts free operation and 50% higher capacity for type II gas achieved with no changes to cryopump external geometry are presented and discussed.

[1]. J.Hutchinson et al. *Mixed mode cracking in layered material*. Advances in applied mechanics, 29, 63 (1992).

[2]. A.J.Bartlett et al. *Pressure burst free high capacity cryopump*. United States Patent Application 20080168778, (2008).

11:00am **VT-MoM9 Combination of Compact NEG and Small Ion Pumps for UHV Systems**, ***P. Manini***, SAES Getters, Italy, *C.D. Park, S.M. Chung*, Pohang Accelerator Laboratory, South Korea

Achieving a better base pressure and reducing bake-out time are the two important practices for an UHV system. Use of a sputter ion pump (SIP) in combination with non-evaporable getter (NEG) is one of the good solutions for this. Although many efforts have been made showing results of the pumping performances of NEG-SIP combination, the SIPs used were relatively large. Furthermore there is a demand for high performance, compact combination pumps that can be installed in a tight space in a storage ring of the proposed PLS-II project. Thus we tested the characteristics of a compact NEG-SIP combination pump (CNP) to see if the CNP can meet the above mentioned desires.

A compact getter cartridge mounted on CF40 flange (Capacitorr D 400-2) was used in combination with small SIPs, having speeds ranging from 10 to 60 l/s. The CNP was attached to a stainless steel chamber that has five CF40 flanges with a total inner surface area of 3,000 $cm^2$.

Base pressures (BPs) of the CNP-UHV system, in a wide range of situations, with/without NEG and with/without baking were measured. Significantly lower pressures and faster pumping could be achieved using the CNP. Base pressures of low $10^{-11}$ mbar could be obtained with a compact NEG for 10 l/s and 60 l/s SIPs after a 48-h bakeout.

The results also show that the compact CNP can provide high pumping speed and reach $10^{-11}$ mbar after a very short (few hours) bakeout. The BP was $1x10^{-10}$ mbar with 60-l/s SIP alone after a 48-h bakeout, whereas it was $7.9x10^{-11}$ mbar with the CNP; a better result after only 2-h bakeout. This is quite a remarkable decrease in the bakeout time of a UHV system.

It is worthwhile to note that UHV could also be achieved with the CNP even in a fully unbaked system: A pressure of $3.9x10^{-10}$ mbar with the CNP was reached, while it was $8x10^{-9}$ mbar with the SIP alone. The other interesting result of the CNP-UHV system is that the pressure increase is much less and slower when the SIP is switched off. This is also a good characteristic, required for portable vacuum devices.

All these characteristics are particularly useful for the design and operation of the vacuum system of a storage ring. It may also be beneficial for the miniaturization of vacuum equipments and mobile applications which require smaller pumping systems.

11:20am **VT-MoM10 Water Vapor Cryopumping: Refrigerant Phaseout Compliance**, ***K. Flynn***, *C. Rebecchi*, Brooks Automation Polycold Systems

Water vapor cryopumps, which use mixed gas refrigeration technology, rely on mixtures containing four or more refrigerants, each with widely spaced boiling points. Historically, these mixtures contained two or more chlorinated refrigerants such as chlorofluorocarbons (CFC's) or hydrochlorofluorocarbons (HCFC's). Both classes of compounds contribute to depletion of the stratospheric ozone layer and are subject to legislative action to phase out these compounds. CFC refrigerants were banned in the US and other developed countries in 1995. HCFC refrigerants are currently in use, but are targeted for phase out. Mixed gas refrigerant water vapor cryopumps have relied on HCFC refrigerants, including HCFC R-22 since the early 1990's when CFC refrigerants were phased out. Although water vapor cryopumps experience much lower leakage rates than commercial refrigeration systems, they are subject to the same laws as all other refrigeration equipment. Effective January 1, 2010, the use of R-22 will be banned on new equipment in the US. R-22 is a key refrigerant in water vapor cryopumps due to its excellent refrigeration capacity and its relatively low freezing point (-160 °C). The phase out of R-22 has required extensive development of alternative refrigerants. It has been accomplished for three important sizes of water vapor cryopumps. The resulting products provide water vapor cryopumping at the same speeds and water vapor partial pressures as the previous mixtures with R-22. This paper reviews the development approach, and compares system and pumping performance for these green products. Experimental data from commercial vacuum systems is presented for the old and new product.

11:40am **VT-MoM11 Vacuum Processing for the 21st Century**, ***S. Ormrod***, *N. Schofield*, Edwards Ltd, UK

On the 90th anniversary of the foundation of Edwards by FD Edwards, the origins of industrial vacuum processing are examined. They are then compared with progress at the time of FD Edwards' death in 1966 and finally, possible developments are examined in the light of vacuum processing technology in 2009.

Proliferation of vacuum processes are shown to drive the evolution from mercury based vacuum technology through oil sealed pumping to dry pumping in the primary and secondary pumping pressure region resulting in a significant market for vacuum processing equipment.

Looking ahead, likely commercial influences are identified, such as electronic, environmental, and safety applications.

To meet that demand, trends and limitations in vacuum engineering are highlighted, particularly capacity, power and rotational speed. The benefits of applying electronics to vacuum processing become apparent - the very products vacuum processing equipment has helped to manufacture so successfully.

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