

Monday Afternoon, October 20, 2008

Vacuum Technology

Room: 205 - Session VT-MoA

Vacuum Cleanliness, Outgassing, Contamination, and Gas Dynamics

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

2:00pm **VT-MoA1 A Review of the Development of Cleaning Processes and Cleanliness Assessment at Daresbury Laboratory.** *J.D. Herbert, K.J. Middleman, R.J. Reid, A.N. Hannah*, STFC Daresbury Laboratory, UK
INVITED

Over the last 30 years the cleaning and processing of vacuum components at Daresbury Laboratory has changed considerably. Although some aspects remain quite similar, others are very different. There are two key reasons for the changes. Firstly, project requirements at Daresbury have changed significantly. Early projects like the Synchrotron Radiation Source (SRS), a 2nd Generation Synchrotron Light Source, required clean Ultra High Vacuum (UHV). Things became more complex when designing DIAMOND, a 3rd Generation Synchrotron Light Source, due to higher photon desorption yields and narrow vacuum chambers. Current projects like ALICE (Accelerators and Lasers In Combined Experiments) and the NLS (Next Light Source) demand vacuum levels in some parts to be in the eXtreme High Vacuum (XHV) region. To achieve this very low levels of contamination are required and very tight restrictions on particles are needed. Secondly, legislation has restricted the range of cleaning processes available. In the early years it was possible to use chlorinated solvents with relative freedom. Environmental legislation then restricted the use of Ozone depleting chemicals and COSHH added further restrictions. Over the last few years both Environmental and Health & Safety legislation has been tightened further. This paper will describe how the vacuum science group at Daresbury Laboratory have adapted to these changes and will describe some of the qualification methods used to determine cleanliness. Also, a recent study of cleaning methods has been conducted, some of the results will be presented and how the results have helped to determine a strategy for the future.

2:40pm **VT-MoA3 The Stainless Steel Bellows with Inner Surface Polishing.** *O. Koizumi*, Osaka Rasenkan Kogyo Co., Ltd., Japan, *N. Ogiwara*, Japan Atomic Energy Agency, *S. Sawa*, Osaka Rasenkan Kogyo Co., Ltd., Japan, *K. Suganuma*, Japan Atomic Energy Agency, *M. Matsue*, Osaka Rasenkan Kogyo Co., Ltd., Japan

One of the performances required for gas feed system in semiconductor manufacturing equipment is to be particle free. To meet this requirement the inner surface of the bellows must be smoothed and cleaned. This problem for the pipe and mechanical processed parts has been already resolved by combination with electro polishing or electro mechanical buffing, etc., and cleaning. However, for the bellows we could polish the inner surface smooth but not evenly because it is formed in bellows shape. Therefore we established wet mechanical polishing with which it is possible to polish the inner surface of the bellows smooth and even. The feature of this polishing is that you can completely polish the entire inner surface of the bellows by mixing abrasive in the liquid. We finally obtained the inner surface roughness less than $Ra=0.05\mu\text{m}$ for the bellows. Furthermore we realized to be particle free by combing this polishing and cleaning. Also we are planning to report the outgassing rate from the bellows which applied this wet mechanical polishing.

3:00pm **VT-MoA4 A Sensor for Detecting Extremely Low Concentrations of Heavy Hydrocarbons in Vacuum Systems.** *N.B. Koster, R. Jansen*, TNO Science and Industry, The Netherlands

At present vacuum systems are becoming more and more complex and are growing towards a full industrial scale. Together with this growth in size and complexity the cleanliness demands are also becoming more strict. Especially for Extreme Ultra Violet Lithography (EUVL) the requirements on hydrocarbon concentration are high. Typically the total concentration of hydrocarbons in such a tool must be below $1*10^{-12}$ mbar integrated over the mass range 45 to 200 in order to maintain the optics quality during lifetime of the EUVL tool. This also means that the requirements for components and sub-assemblies are even more strict and because the tool cannot be baked the cleanliness has to be verified before integration in the tool. These type of cleanliness demands are also becoming more important for other applications like ALD, CVD, fusion and accelerator systems. We present a sensor, that we are currently developing, that is able to measure the total concentration of heavy hydrocarbons (including fluor compounds) from

mass 100 up to infinity in a single channel. The sensor is based on a ionisation source in combination with a magnetic analyser that is used as a low mass filter for removing the species that are of no interest. The masses of interest are led towards a single channel detector for readout of the hydrocarbon partial pressure. Unlike an ordinary RGA the mass range is not scanned but acquired on a single detector, which leads to a significantly higher signal. Also, unlike a RGA, the noise level of the detector is not summed over the scanned mass range, but remains constant. As the sensor is a one channel detector it acts as a Mass Filtered Ion Gauge (MFIG) giving a signal for total concentration of heavy molecules. The benefit of such a sensor is that it gives a single output which enables a go / no go decision for vacuum qualification of chambers and components or in-situ monitoring of contamination levels in a tool. We expect that the sensor has a lower detection limit below $1*10^{-13}$ mbar when operating in faraday mode. The detection limit can be further increased by adding a Secondary Electron Multiplier (SEM) like a Multi Channel Plate (MCP) in front of the faraday detector. This can increase the sensitivity with a factor 100 to 1000. We will present our first results of the sensor when operating at vacuum levels between $1*10^{-6}$ and $1*10^{-9}$ mbar.

3:20pm **VT-MoA5 Low Outgassing, Low Permeability Elastomeric Seals for High Purity PVD Processing.** *G.A. Foggiato, W.B. Alexander*, Greene Tweed Co.

New processes associated with semiconductor manufacturing and in related industries require very clean, high vacuums. Systems utilized for such processes employ a multitude of seals with emphasis in elastomeric seals for transfer handling of substrates. The process environments require such seals to have very low outgassing and low permeability to the atmosphere as well as the gases used in processing. Recent developments of new elastomer seals employing perfluoroelastomers provide the characteristics suitable for these applications. Various material and fabrication technologies are described as used for fabricating seals for high vacuum, ultra high purity environments. This paper will review the new technologies and their effect on the properties of seal materials, the seals themselves and associated equipment performance. Examples of such optimization of seal technology will show the achieved characteristics for seals capable of sustaining vacuums of 10^{-9} Torr and at temperatures to 280 °C. The outgassing characteristics of a variety of seals will be shown for H₂O, HF, H and O₂, all potential contaminating gases during depositions of films for semiconductor and photovoltaic thin films. The low level of outgassing attainable by the more recently developed materials and the technology associated with such properties will be described. Through the use of a "tighter" bonding perfluoro molecular structure, combined with molecular based fillers, a molecular structure has been developed which greatly reduces permeability. Through a uniquely developed molding process, key of which is the cooling cycle, a tighter molecular structure is achieved. These new materials provide both the characteristics required for high performance PVD deposition equipment as well as addressing the highly corrosive and aggressive environments found in semiconductor etching and CVD equipment. The progress made in increasing the longevity of seals in such applications is described as this is becoming very critical in future technology nodes manufacturing. Data for use with NF₃, CF₄ and O₂ plasmas is presented to better understand the deterioration of the elastomers and associated mechanisms. The impact of temperature is also described showing that at chamber temperatures approaching 300 °C, such new seal materials still function suitably. Descriptions of these extended applications will be given along with the resultant enhanced performance.

4:00pm **VT-MoA7 Novel Leak Testing Methods for High Reliability Hermetic Devices.** *J.M. Hochrein, S.M. Thornberg, J.R. Brown, M.I. White*, Sandia National Laboratories

Traditionally, leak testing is done using helium leak detectors, which are specially designed for the detection of helium. Helium leak tests are often limited to room temperature which does not allow information to be gathered at elevated temperatures. This type of test only provides information about the leak rate under ambient conditions and not about the temperature at which a leak occurs during a thermal profile or how the leak magnitude changes as a function of temperature. In other cases, components may be sealed and contain gases that cannot be detected using traditional techniques rendering them ineffective. Two newly developed experimental methods for leak testing high reliability hermetic devices will be discussed. The first method is used to conduct real-time leak monitoring at temperatures ranging from ambient to 900°C while subjecting one side of the parts to continuous vacuum and monitoring for air components. Any loss of hermeticity will result in increases in air components detected. The second method that will be discussed is a method that involves monitoring for the presence of a specific organic target molecule (in this case, a

perfluorinated hydrocarbon) to localize the source of a leak. These experiments are conducted at ambient pressure using solid phase micro extraction (SPME) combined with gas chromatography/mass spectrometry.

4:20pm VT-MoA8 Direct Conductance Measurements of Small Leaks with Simple Geometries, J.A. Fedchak, R.F. Berg, D.R. Defibaugh, National Institute of Standards and Technology

The Pressure and Vacuum Group at NIST utilizes two constant pressure flowmeters as primary standards to calibrate helium leak artifacts, spinning rotor gauges, and ion gauges, using an orifice flow technique. A molar flow rate is produced by allowing gas to leak out of a small volume through a leak valve. By driving a piston into the volume such that the pressure within the volume remains constant, the gas flow rate through the leak valve is determined from the pressure measurement times the volume displacement of the piston per unit time (L/s). In the absence of other sources or sinks of gas, the conductance of the leak is identical to the volume rate of change of the piston. Therefore, the NIST flowmeters can be directly used to measure the conductance of leaks in the range of 10^{-6} to 10^{-5} L/s. Similar measurements have been done by other labs with primary flow standards (see Jousten et al.,¹ for example), but with limited success due to the inability to accurately model the conductance of leaks with complicated geometries. We directly use the flowmeter to measure the conductance of small leaks with simple geometries, such as thin orifices (OD~10 μ m) and capillaries. Small leaks with a known, precisely measured conductance may be used as check standards and in a variety of other applications. The conductance measurements and the applications will be presented.

¹ K. Jousten, H. Menzer, and R. Niepraschk, *Metrologia*, 39, 519 (2002).

4:40pm VT-MoA9 Modeling of Diffusion Processes in Vacuum Technology with Finite Element Method, J. Setina, Institute of Metals and Technology, Slovenia

Diffusion of gasses is a frequent phenomenon in vacuum technology. Examples are: permeation of atmospheric gasses through parts of chamber wall (elastomeric seals and plastics), diffusion of dissolved hydrogen gas in a chamber wall and its evolution into vacuum, and permeation of gasses through membranes in permeation leak elements. The process can be mathematically represented with a diffusion equation, which is a second order partial differential equation. Its solutions depend on the geometry of the system and on initial and boundary conditions. Solutions can be found in analytical form for simple geometries and selected simple initial and boundary conditions only. Diffusion equation can be solved also numerically with finite element method (FEM). Numerical calculations can be applied for arbitrary initial and boundary conditions. In the presentation we will discuss a simple one dimensional case, which can be applied when the problem can be approximated with planar geometry. Numerical calculation of a time dependency of the concentration profile within the sample can be easily performed in a widely use spreadsheet program Excel. The reliability of FEM calculations was checked for a special case of initial and boundary conditions, when the analytical solution is known also. We will also present a practical example of FEM modeling of He gas flow into a vacuum system through a Viton gasket after short-time exposure to He at atmospheric side. Such problem is often observed when He leak detection is performed.

5:00pm VT-MoA10 Rarefied Gas Flow through a Slit into Vacuum, F. Sharipov, Universidade Federal do Parana, Brazil, D. Kozak, Pontificia Universidade Catolica do Parana, Brazil

The rarefied gas flow through a thin slit represents a great practical and scientific interest. Such kind of flow takes place in many engineering applications, e.g. vacuum equipment, microfluidics, spacecraft design, metrology of gas flow etc. In the present work, such a flow is studied, based on the direct simulation Monte Carlo method. The mass flow rate is calculated with an accuracy of 0.5% over the whole range of the gas rarefaction, from the free molecular regime to viscous one. The flowfield is also provided.

5:20pm VT-MoA11 Moving Surfaces in DSMC: Implementation, Validation and Applications, R. Versluis, M.E. Roos, L. Thielen, TNO Science and Industry, The Netherlands

Applications in vacuum technology exist where moving surfaces play a role. Under rarefied conditions, moving surfaces influence heat and momentum transfer and surface stress. Several methods have been used to model moving surfaces in Test Particle and Direct Simulation Monte Carlo methods. In simple situations, such as plane Couette flow, one can add an extra velocity vector to molecules hitting a moving surface (the modeled surface stays at rest in an inertial domain). In other examples the flow field is simulated by using a moving calculation domain and the molecule trajectory is corrected for this. Both methods are limited to only one moving surface. But in cases where the domain contains both moving and non-

moving surfaces these methods cannot be applied or separate domains need to be defined and the interaction at the interface needs to be prescribed or iteratively determined. We have developed an algorithm to simulate an arbitrary number of moving and non-moving planes in one domain for DSMC methods. The planes can have any velocity vector but should not change the geometry. The method uses periodic boundary conditions (especially implemented for this) and is grid independent. In the DSMC scheme molecule trajectories are determined in two steps: particle movement between collisions and particle collisions. Boundary interaction is taken into account during the particle movement phase. In our method the moving surface is a special boundary with all properties of a normal boundary (temperature, accommodation coefficient, reflection velocity distribution). The difference is that the trajectory of each surface is determined and the exact time and place of interaction between the boundary and a molecule is determined. This way, the exact molecule trajectory is determined during its movement phase taking into account the plane movements during that time. Multiple collisions in one time step between moving and non-moving surfaces are taken into account. The current algorithm is limited to 2D with planes moving in a straight line, but the principle is valid for 3D and planes moving with arbitrary and changing velocities (although the calculation of collisions between plane and molecules becomes more tedious). Results will be shown of validation simulations and possible applications, such as simulations of displacement pumps, moving stages or surfaces in lithography, CVD, PVD and ALD systems, sample manipulation, vacuum conveyor belts etc.

Authors Index

Bold page numbers indicate the presenter

— A —

Alexander, W.B.: VT-MoA5, 1

— B —

Berg, R.F.: VT-MoA8, 2

Brown, J.R.: VT-MoA7, 1

— D —

Defibaugh, D.R.: VT-MoA8, 2

— F —

Fedchak, J.A.: VT-MoA8, 2

Foggiato, G.A.: VT-MoA5, 1

— H —

Hannah, A.N.: VT-MoA1, 1

Herbert, J.D.: VT-MoA1, 1

Hochrein, J.M.: VT-MoA7, 1

— J —

Jansen, R.: VT-MoA4, 1

— K —

Koizumi, O.: VT-MoA3, 1

Koster, N.B.: VT-MoA4, 1

Kozak, D.: VT-MoA10, 2

— M —

Matsue, M.: VT-MoA3, 1

Middleman, K.J.: VT-MoA1, 1

— O —

Ogiwara, N.: VT-MoA3, 1

— R —

Reid, R.J.: VT-MoA1, 1

Roos, M.E.: VT-MoA11, 2

— S —

Sawa, S.: VT-MoA3, 1

Setina, J.: VT-MoA9, 2

Sharipov, F.: VT-MoA10, 2

Suganuma, K.: VT-MoA3, 1

— T —

Thielen, L.: VT-MoA11, 2

Thornberg, S.M.: VT-MoA7, 1

— V —

Versluis, R.: VT-MoA11, 2

— W —

White, M.I.: VT-MoA7, 1