Vacuum Technology Division
Room 203B - Session VT-MoM

Vacuum Measurement

Moderators: Marcy Stutzman, Jefferson Lab, Alan Van Drie, TAE Technologies

8:20am **VT-MoM-1 Pharmaceutical Freeze-Drying and Vacuum-Drying: Challenges and Opportunities**, ***Evgenyi Shalaev,*** Allergan **INVITED**

Many drugs are unstable in aqueous solutions, and drying is commonly used to improve their storage stability and shelf life. Freeze-drying is the most common drying method for parenteral pharmaceutical dosage forms, including both small molecular weight drugs and biologicals. Alternative vacuum drying technologies have also been introduced, although predominantly for research and development purposes. The presentation focuses on freeze-drying, starting with a brief overview of lyophilized (freeze-dried) products and corresponding manufacturing processes. The importance of pressure control during all three stages of freeze-drying (i.e., freezing, primary drying/ice sublimation, secondary drying / desorption of non-frozen water) is emphasized.

9:00am **VT-MoM-3 Fixed Length Optical Cavities for Primary Traceability to the Pascal**, ***Jay Hendricks,*** *J Ricker, K Douglass,* National Institute of Standards and Technology; *G Brucker, E Fuchs, D Ocepek, P Sullivan, S Venkatesan,* MKS Instruments, Inc., Pressure and Vacuum Measurement Group

Over the past 5 years, NIST has worked to develop a new pressure standard based on the fundamental properties of gas refractive index that will replace mercury manometers at national metrology institutes and has potential to be developed as a commercially manufactured product. The new pressure standard is based a first-principles quantum-chemistry calculations of gas refractive index and is a new route to realizing the pascal. NIST has now built and tested a fixed-length optical cavity (FLOC), which consists a pair of Fabry–Pérot cavities within a single block of ultralow-expansion glass. The change in optical path length between the two cavities (one at vacuum and one at the pressure to be measured) depends on the gas refractive index, density, and atomic or molecular properties. Helium's atomic properties were calculated from first principles, so the refractivity measurement leads to a determination of density, which provides a determination of pressure. While helium's refractive index has now been calculated by theory, the value of nitrogen refractive index remains too difficult for current computational theory to handle. Using the NIST mercury manometer along with helium's theoretical value of refractive index in a FLOC has resulted in a new experimental value for nitrogen refractive index to be determined. This enables the FLOC to be used with nitrogen as a pressure standard with direct primary traceability to NIST. Moving forward, the FLOC technology is so promising as a pressure standard, that NIST has joined with MKS under a Collaborative Research and Development Agreement (CRADA). The aim of this partnership is technology transfer to the market place, with the aim to develop a small, portable prototype need for real world metrology operations for industrial applications in gas pressure metrology. The current status of NIST-MKS CRADA will be briefly presented and discussed.

9:20am **VT-MoM-4 Fundamental Quantum-based Vacuum Metrology at NIST**, ***Julia Scherschligt,*** National Institute of Standards and Technology

NIST has developed and characterized a variety of vacuum standards over the last several decades. Much effort, particularly recently, has been placed into developing standards based on optical methods and fundamental quantum properties. In this talk, I will present an overview of these efforts, focusing on the more recent advances in vacuum metrology. These span a wide range of pressures and employ a variety of nascent methods. However, our most recent methods focus on developing absolute standard based on fundamental physical properties, particularly quantum properties. At the low vacuum, we probe the pressure-dependent index of refraction of a gas in a fixed-length optical cavity (FLOC). At the middle range from the viscous flow regime to the high vacuum, we relate the ring-down time of a membrane to pressure ("Brane" gauge). At the ultra and extreme high vacuum (UHV and XHV), we use the loss-rate of ultra-cold atoms from a magnetic trap to measure background particle energy density in the cold-atom vacuum standard (CAVS). Each of these techniques presents unique technical challenges, I will put these challenges in context and briefly describe the research ongoing to address them. These include techniques to measure the refractivity of gases and distortion characterization for the FLOC, optomechanics and nanophotonics for the Brane gauge, and collision cross section measurements for the CAVS.

9:40am **VT-MoM-5 Moving the FLOC to the Telecom**, ***Kevin Douglass,*** *J Ricker,* National Institute of Standards and Technology; *J Hendricks,* National Institute of Standards and Technology (NIST)

Towards the goal of quantum based traceability of the SI, NIST has developed an optical pressure standard where traceability is achieved through accurate quantum mechanical calculations of the refractive index and virial coefficients of helium. To achieve widespread adoption of this novel optical pressure measurement technology we leverage the various technologies that have been developed to support the telecommunications industry. We have begun characterizing the performance of our Fixed Length Optical Cavity (FLOC) at 1542 nm. At this wavelength an acetylene stabilized laser can be used to measure the wavelength to better than a ppm, which is one of the requirements of the measurement. The new optical setup and methodology for achieving high accuracy will be discussed along with future challenges and a detailed look at the sources of uncertainty and methods for calculating pressure from the change in refractive index.

10:00am **VT-MoM-6 Transient Method of Permeability Measurements for Microporous Media**, *M Johansson,* Aix Marseille University, France; *M Wuest,* INFICON, Liechtenstein; *P Perrier,* ***Irina Graur Martin,*** Aix Marseille University, France

The gas flow through the low permeable porous media have a great interest, especially in vacuum technology for filtering, separation process, protection and flow control. It can combine high mass flow rate and a high level of rarefaction. This property makes it particularly suitable as a leak element, by taking advantage of the constancy of conductance in free molecular regime, for example for calibration of ionization gauges or mass spectrometer [1]. The transient experimental technique, developed previously for the mass flow rate measurements through the microchannels [2], is generalized to obtain the permeability directly from the pressure variation measurements. The present experimental methodology, allowing for step by step data verification, leads to higher accuracy than the similar and commonly used method such as "pulse-decay" techniques [3]. The measured data are fitted according to the exponential function with the pressure relaxation time as a single fitting parameter. The new expression for the permeability is proposed involving besides of the geometrical parameters, the ratio between the gas relaxation time (inverse of the gas collision frequency) and the pressure relaxation time. The permeability of the microporous media with the characteristic pore size of 0.2 and 0.5 microns is measured for different gases. It was found that the permeability at low pressure (3 Torrs) increases 50 times compared to atmospheric pressure permeability . This permeability increasing depends essentially on the gas nature.

References:

[1] Hajime Yoshida, Kenta Arai, Hitoshi Akimichi, and Tokihiko Kobata. Newly developed standard conductance element for in situ calibration of high vacuum gauges. *Measurement,* 45(10):2452 – 2455, 2012. Special Volume.

[2] M Rojas Cardenas, I Graur, P Perrier, and J G Meolans. Thermal transpiration flow: a circular cross-section microtube submitted to a temperature gradient. *Phys. Fluids,* 23:031702, 2011.

[3] W. F. Brace, J. B. Walsh, and W. T. Frangos. Permeability of granite under high pressure. *Journal of Geophysical Research,* 73(6):2225–2236, 1968.

10:40am **VT-MoM-8 Beamline Technology and Current Modeling Capabilities for Ion Implantation**, ***Svetlana Radovanov,*** Applied Materials, Varian Semiconductor Equipment **INVITED**

Ribbon beam technology have been used in semiconductor ion implantation for past three decades. Over the years these ion implanters have become highly sophisticated tools incorporating the use of energy filters, collimators, quadrupoles, scanning systems and more recently molecular plasma sources, cryogenic and elevated implant temperature capabilities. One of the features that made these tools so successful in device fabrication is the high degree of control of the dopant depth profile. By selecting a unique ion mass, ion charge, ion energy and implant angle, a beam line tool offers highly automated control over the beam transport and implanted ion dose [1]. These beam lines operate with a large variety of species, several orders of magnitude energy and dose range. The wafer processed per hour reach 500 wafers/hour for a standard high current implanter. In recent years, some very high dose applications have been enabled by plasma doping systems [2]. For example, some dynamic random-access memory applications require incredibly high doses ~$5x10^{16}$ /cm^2 that can be done by plasma doping systems. Unlike the beam line tools, ions are not mass analyzed, but instead the wafer is processed within the plasma chamber or in an adjacent vacuum chamber. The wafer is pulsed negatively by a bias supply with a square wave T~50ms and f~5-50 kHz. Implant energy is controlled by the bias voltage which can exceed 10 kV. The plasma is generated by an inductively coupled rf coil. When the bias voltage is on, a plasma sheath forms in front of the wafer surface, across which ions are accelerated and are implanted into the silicon.

In this paper, we will discuss electrostatic focusing, filtering and steering of an ion beam and modeling associated with it. This will include low energy beam acceleration, deceleration and transport. We also describe the 2D and 3D codes that are used to model beam line optical elements.

REFERENCES

A. Renau, Review Scientific Instruments, 81, 02B907 (2010)

J. England and W. Moller, Nucl. Inst. Methods, 365, 105 (2015)

11:20am **VT-MoM-10 Design of a New Thermal Vacuum Chamber for Space instrument Calibration***, **Freek Molkenboer**, R Jansen, R Veraar, G Otter, W van Werkhoven, N Koster, F Driessen,* TNO, Netherlands

TNO is investing in a new facility for calibration of optomechanical Space instruments. This facility, called Calibration Space Instruments (CSI) should be operational early 2021. To meet this deadline the conceptional design phase has started early 2018.

The facility has three major sub system; a Thermal Vacuum Chamber (TVC), an Optical Ground Support Equipment (OGSE) and a Mechanical Ground Support Equipment (MGSE).

The OGSE system will provide all the optical stimuli that are required to perform an optical calibration of a Space instrument.

During a calibration of a Space instrument many relative positions between the OGSE and the instrument must be tested. The MGSE is responsible for the high accuracy, and highly reproducible manipulation of both the OGSE and the instrument. It is expected that some of the manipulation is done in vacuum, leading to the corresponding challenges.

The calibration of the instrument must be performed at the temperature in which it will operate in orbit. The TVC needs to provide these conditions. Beside the operational temperature the instrument also needs to be tested at non-operational temperatures, which increases the temperature range. It can be expected that some parts of the instrument will require LN2 temperatures. The CSI facility will focus on calibration of mid-size instruments, this results in a chamber with a volume up to 15 cubic meter

The vacuum pressure during a calibration shall be below 10-5 mbar. The challenge is that the materials used in a Space instrument and the TVC absorb a lot of water when exposed to air, resulting in a high pumping speed needed to reach the required pressure.

Future Space instruments will have higher resolution, which will directly impact the calibration facility. To be able to perform a calibration, pointing accuracies of 0.0015° are needed, which might result in for instance active shielding of vibrations from the TVC system and the floor towards the instrument and the OGSE.

Space instruments represent a lot of money, therefore instrument safety is crucial in the design of the facility.

During the oral we will discuss challenges that come with the design of the TVC for Space instrument calibration, and the measures that are taken to ensure safe and successful calibration campaigns.

11:40am **VT-MoM-11 Pressure Measurements from Combining Non-evaporable Getter Pumps and a Novel Extreme High Vacuum Cryopump***, **Marcy Stutzman**,* Thomas Jefferson National Accelerator Facility*; A Segovia Miranda,* Universidad Aut´onoma de Zacatecas*; P Adderley, M Poelker,* Thomas Jefferson National Accelerator Facility

The Jefferson Lab polarized electron source requires vacuum approaching extreme high vacuum for long operational lifetime for the GaAs photocathodes. Currently the system is pumped with a combination of non-evaporable getter (NEG) pumps, ion pumps, and a NEG coating on the chamber walls. Exploring further improvement of the vacuum for the system, we have assembled a system using an array of NEG modules and a novel cryopump with Boron Nitride Nanotubes (BNNT) instead of the traditional charcoal. The BNNT has been mechanically attached to the cryosorption surfaces of a commerical cryopump, and the system fully baked to remove water with no adhesive present in the system. We report here on the pump speed of the BNNT cryopump, and characterize the base pressure achieved in the combined NEG/cryopump system using both an extractor gauge and a Watanabe 3BG XHV ionization gauge which has reached at least the x-ray limit of the extractor gauge.

Author Index

Bold page numbers indicate presenter