

# Monday Afternoon, October 30, 2017

Vacuum Technology Division  
Room: 7 & 8 - Session VT-MoA

## Material Outgassing, Adsorption/Desorption and XHV

Moderators: Giulia Lanza, SLAC National Accelerator Laboratory, Jacob Ricker, NIST

1:40pm **VT-MoA1 Weight of Water on the Solid Surface in Air and Vacuum**, *Richard Green*, National Research Council of Canada, Canada **INVITED**

In 2018, after over 30 years of research by national measurement laboratories around the globe, the unit Kilogram is expected to be redefined in terms of a fundamental constant of nature; Planck's constant. The present definition has not significantly changed in over 120 years and relates to a single cylinder made of exactly one kilogram of platinum-iridium alloy that is stored in a vault in Sèvres, France. In order to connect the present Kilogram measured in air to a redefined Kilogram measured in vacuum, new tools and methodologies have been developed to understand and quantitatively determine the change in mass that occurs when metals are placed in vacuum. While initially concerned with platinum, work has extended to stainless steel and other surfaces. With resolutions on the order of a 100 parts per trillion possible, sorption of less than 0.01 monolayers of water is observable.

In this talk we will present measurement techniques and tools used to quantitatively and traceably determine the weight of water and hydrocarbons desorbed from a surface as it is exposed to vacuum. The techniques have been used to study factors such as pressure, surface roughness and contamination, which influence the quantity and dynamics of desorbed mass. The investigations will be presented in context of efforts at the National Research Council of Canada to make the world's most accurate measurement of Planck's constant.

2:20pm **VT-MoA3 Hydrogen Measurement using a Thermal Desorption Spectrometer**, *JongYeon Lim*, Korea Research Institute of Standards and Science, Republic of Korea, *K.D. Kim, H.S. Oh, C.H. Lim, Y.D. Joh*, Infinity Vacuum Technology, Republic of Korea

The TDS system, designed for measuring outgassing rate of a diameter of 10 mm sample, with two pumping paths; one is ordinary throughput path with an orifice, the other a UHV path, has been newly developed to measure any molecules on samples with a sophisticated DAQ system.

Two paths are directly connected to the main chamber equipped with devices including a rod-guided halogen heater allowing the sample temperature up to 1200 °C .

The throughput path utilizes the UHV equipment to measure the outgassing rate quantitatively and qualitatively. In the case of quantitative hydrogen measurement, the throughput path does not have enough pumping speed since a small orifice diameter has the conductance limitation.

The experimentally acquired system calibration factor for the throughput method is 16.0, which is defined as the ratio of background outgassing rates with the gate valve closed and open using the throughput method.

In order to verify the measurement reliability a NIST Standard Reference Materials (SRM) was introduced to the TDS system. Most of hydrogen was desorbed during the course of heating process up to 800 °C for 90 minutes. The area under the H<sub>2</sub> peak is proportional to the nominal value of 126.8 wt ppm (uncertainty of 2 %). The hydrogen calibration factor of 4.38686E23 was realized. The system claims the hydrogen measurement resolution of 1.3E-6 wt ppm.

In this presentation we briefly introduce the accurate UHV outgassing measurement system for both qualitative and quantitative analyses, which has an 18 % uncertainty of total outgassing rate with Inficon BPG400 HV gauges and a unique self-calibration function.

*Acknowledgements: Results are partially attributed to two national projects sponsored by the Korean Ministry of Trade, Industry & Energy, and the KRISS main project (Contract Nos. 10048806, and 170111649).*

2:40pm **VT-MoA4 Automatic Flowmeter and Dynamic Expansion System for UHV/XHV Studies**, *James Fedchak, J. Scherschligt, D. Barker, S. Eckel*, NIST

NIST is presently creating the Cold Atom Vacuum Standard (CAVS), a quantum-based fundamental primary vacuum sensor which significantly departs from present methods of measuring and realizing ultra-high vacuum

(UHV) and extreme-high vacuum (XHV). The CAVS is an absolute sensor based on the loss-rate of ultra-cold atoms from a conservative magnetic trap due to collisions of the trapped cold atoms with the ambient background gas, and will cover a pressure range of 10<sup>-8</sup> torr to below 10<sup>-12</sup> torr, thus spanning UHV and into XHV. Knowledge of the thermalized collision cross-section, or loss-rate coefficient, between the trapped ultra-cold sensor atoms and the background gas is critical to operation of the CAVS; such collisions are also important in other experiments and devices based on trapped cold atoms because they limit the lifetime of atoms in the trap and, in addition, glancing collisions can increase statistical and other uncertainties. To support the CAVS and other atomic physics programs, NIST is developing a dynamic expansion system to set a known pressure in the CAVS, which will allow the experimental determination of collision cross-sections, relative gas-sensitivity factors, and facilitate studies of other systematic effects. To that end, we are developing a constant pressure flowmeter capable of producing flows of at least 10<sup>-13</sup> mol/s and a dynamic expansion system to produce pressure rises as small as 10<sup>-10</sup> Pa. The system is designed to produce low gas-flows of H<sub>2</sub> and many other gases of interest, to be fully automated, and to have extremely low outgassing rates. The design and construction of the UHV/XHV flowmeter and dynamic expansion system will be discussed.

3:00pm **VT-MoA5 Development of a New UHV/XHV Pressure Standard (Cold Atom Vacuum Standard)**, *Julia Scherschligt, J.A. Fedchak, S. Eckel, D. Barker*, NIST **INVITED**

NIST has a long history of laser cooling and trapping of neutral atoms, largely motivated by building better time standards or clocks, and has recently begun a program to extend the metrological capabilities of cold trapped atoms to measurement of vacuum. This will align vacuum metrology to the emergent NIST "Quantum SI" paradigm, in which a measurement has intrinsic traceability and the line between sensor and standard is blurred. Since the earliest days of neutral atom trapping it has been known that the background gas in the vacuum limits the lifetime of atoms in the trap. We are inverting this problem to create a quantum-based standard and sensor. Indeed, because the measured loss-rate of ultra-cold atoms from the trap depends on a fundamental atomic property (the loss-rate coefficient or thermalized cross section) such atoms can be used as an *absolute* sensor and *primary* vacuum standard. Researchers have often observed that the relationship between the trap lifetime and background gas can be an indication of the vacuum level, but a true absolute sensor of vacuum has not yet been realized. This is because there are many technical challenges that must be overcome to create a device that's truly absolute and primary. The NIST program addresses these challenges both theoretically and experimentally: we have begun *ab initio* calculations of collision cross sections between the trapped cold atoms and the background gas and, on the experimental side, we are thoroughly investigating the systematic uncertainties associated with using an atom trap to determine vacuum level, particularly those associated with loss mechanisms (in a non-ideal trap) other than due to background collisions. We are designing and building the apparatus to measure relevant cross sections, and building our first prototype vacuum sensing apparatus. In this presentation, we will discuss our theory progress, and present our newest measurements, as well as discuss how the Cold Atom Vacuum Standard fits into the broader picture of the NIST dissemination of the Quantum SI.

4:00pm **VT-MoA8 VTD Early Career Award Invited Talk: Modern Metrology Practice for Calibration and Reliability Testing of Vacuum Measurement Products**, *Scott Heinbuch\**, MKS Instruments, Inc. **INVITED**

Vacuum technology is traditionally very slow moving in terms of new techniques and innovations. The measurement techniques of today don't differ drastically from 20 years ago or even longer. That does not mean there aren't new ways we can think about how we use that measurement technology in a metrology lab today. Often times, critical vacuum measurements take time; time being a taboo word in today's product development discussions. There are many practices to help us reduce time waste while still ensuring good vacuum measurement. Principles of Lean have helped us to reduce types of waste in our lab creating an efficient environment where experiments can be set up accurately and timely. Similarly, Six Sigma ideas have given us a set of tools and a common language to quickly identify root cause for problems and simplify our data analysis techniques. These same tools have helped us to identify critical parameters for measurement systems analysis and better understanding of our measurement uncertainties when making measurements compared to our best in class Spinning Rotor, Stabil-Ion®, and Baratron® reference transducers. To further simplify our lab experience, vacuum system complexity has been reduced by an effort to separate the vacuum control system from our test

\* VTD Early Career Award

development in which any user can interface with a vacuum system in the same way they would interface with a common laboratory instrument whether manually, or through a programming interface. Finally, none of this matters unless we are making our measurements with our customers in mind. Several customer applications have been reproduced and turned into standard tests for our products to improve our customers experience and our overall product performance and reliability.

**4:40pm VT-MoA10 Outgassing Rate Measurements of New Materials at NIST, Makfir Seifa, J.A. Fedchak, J. Scherschligt, A. Zeeshan, NIST**

The Thermodynamic Metrology Group at NIST is interested in investigating the outgassing rates and gas uptake properties of a variety of materials. The motivation of these investigations is to support programs aimed to develop new vacuum, pressure, and temperature sensors. For example, materials with ultra-low outgassing rates are necessary to develop a cold-atom vacuum standard (CAVS), which is a metrological-quality XHV/UHV sensor based on ultra-cold atom technology. Chambers and vacuum components used in the CAVS must have ultra-low outgassing rates to achieve UHV and XHV backgrounds. The group is also interested in developing embedded temperature and pressure sensors, where the outgassing or gas uptake properties could influence sensor performance or applicability. In addition, the gas uptake properties of unique materials can be exploited for gas sensors or storage. This talk will highlight the outgassing measurements of heat-treated stainless steel chambers and 3-D printed stainless steel and titanium chambers. In addition, absorption and desorption of gases in 3-D printed nano-composite plastics will be discussed.

**5:00pm VT-MoA11 Scaling up an Ion Implant Process Chamber Cryopumping for 450mm Wafer Processing, Steve Borichevsky, Applied Materials, Varian Semiconductor Equipment**

The semiconductor industry recently explored scaling up the wafer diameter from 300mm to 450mm. Ion Implant faced the challenge of providing process vacuum conditions for larger dopant ion beam currents and coping with the outgassing cause by the ion beam striking increased area covered by photoresist. The process chamber, where the ion beam strikes the target wafer, posed the most difficult vacuum challenges. The increased wafer size was predicted to generate 2.25 times the normal gas loads which would require nine 320mm cryopumps. In order to meet the requirements of implant, three prototype 500 mm diameter cryopumps were mounted onto a process chamber and tested. This presentation describes the basic architecture of an ion implanter, the decisions that lead up to the use of 500mm cryopumps, the results of the initial vacuum system testing and the Monte Carlo simulations.

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