## Tuesday Afternoon, November 8, 2022

#### Vacuum Technology Division Room 301 - Session VT-TuA

#### Vacuum Pumping, Leak Detection, and Modeling

Moderators: Jason Alfrey, Vacuum Technology, Inc., Freek Molkenboer, TNO Science and Industry, the Netherlands

2:20pm VT-TuA-1 Design and Fabrication of Ultra-High Vacuum Test System for Quantitative Determination of Hydrogen Gettering and Permeation of Various Materials, *Ewa Ronnebro*, *R. Storms*, *S. Suffield*, Pacific Northwest National Laboratory; *M. Boeckmann*, *A. Parrot*, *J. Alfrey*, Vacuum Technology, Inc. INVITED We will discuss a recently built state-of-the-art ultra-high-vacuum (UHV) test system with sensitive detection and quantification of hydrogen uptake, solubility and diffusion in various materials. The test system's manifold is equipped with capacitance diaphragm gauges (CDG), residual gas analyzer, spinning rotor gauge, calibrated volumes, turbo pumps, scroll pumps and a leak detector. The manifold is surrounded by a bake-out oven to keep

impurity levels sufficiently low. Two sample chambers are enclosed by high-temperature furnaces. The design was developed by Pacific Northwest National Laboratory (PNNL) in collaboration with Vacuum Technology Inc (VTI). This automated UHV system can be used for several studies of hydrogen-metal interactions including absorption/desorption kinetics, thermodynamics, isotherms, plateau pressures, isotope studies, gaseous impurity identification and permeation rate.

#### 3:00pm VT-TuA-3 Gas Partial Pressure Measurement by Remote Plasma Optical Emission Spectroscopy & Automated Analysis Using Artificial Intelligence, *Dermot Monaghan*, J. Brindley, B. Daniel, V. Bellido-Gonzales, Gencoa Ltd, UK

Vacuum deposition processes are being equipped with an ever-expanding array of sensors to gain more control over the process conditions. Unfortunately, this often presents the operator with too much data to be able to draw clear insights into the performance of the process. Machine learning algorithms are a powerful tool for analyzing large and complex sets of data and have been at the forefront of a revolution artificial intelligence. These techniques are ideally suited for analyzing problems encountered in vacuum processes, which are often expressed as "classification problems", i.e., identifying if a leak is present in the system or not. In particular, they can be applied to the automated analysis of vacuum processes by remote plasma optical emission spectroscopy (RPOES).

Remote plasma OES provides critical information on the state or condition of a process via measurement of residual gas partial pressure present in the chamber. RPOES is now a popular method of residual gas analysis (RGA), as it is industrially robust compared quadrupole mass spectrometry methods and operates from 0.5 mbar to 10<sup>-7</sup> mbar. Whilst RGA information is important, expert knowledge is often required to be able to interpret the data, and in some cases, the spectra are too complex to extract key information using the human eye alone. This paper will present the application of a machine learning A.I. to the automated analysis of magnetron and remote plasma OES data. Examples include leak detection, organic contamination detection and the identification of organic molecules from cracking patterns.

# 4:40pm VT-TuA-8 How Vacuum Controlled Venting Can Improve the Imagery of Electron Microscopy, *Tim Collins*, DigiVac

DigiVac was recently approached to assist a customer with vacuum control in an electron microscope. Most electron microscopes are high-vacuum instruments, as vacuum is needed to prevent arcing and to allow the electrons to travel within the instrument unimpeded. However, the specific microscopes used by this customer have no on-board vacuum control. The user chooses between Low Vacuum and High Vacuum settings depending on the makeup of the sample being observed.

The customer observed that when using the High Vacuum setting, a vacuum level within the microscope deeper than 20 Pascals (approx. 150 millitorr) caused visible degradation in the final image. They emphasized their need to precisely control the vacuum between 20 and 30 pascals for optimal imaging. We hypothesized that raising the pressure slightly, then

maintaining it, would attract the negatively charged electrons causing imaging difficulty.

Our engineering team designed a comprehensive solution using our FYRA Bleed Vacuum Controller allowing ambient air (or a connected gas supply) to be drawn into the chamber as the vacuum pump evacuates it, "balancing" the vacuum level where the user specifies. The bleed valve opens and closes using feedback control based on the current chamber pressure, measured with a separate thermocouple sensor.

Our experiments with the customer's electron microscope showed obvious image degradation sub-20 Pascals with significant improvement when the vacuum level was strictly controlled between 20 and 30 Pascals. After some fine tuning with the built-in PID controller that is included with FYRA, we had a clearly defined SOP for alleviating the degradation issues when the vacuum level dipped below the required range. This type of bleed valve technology has a wide range of scientific applications not just in electron microscopy, but for any user looking to precisely control vacuum, introduce gasses during processing applications, or improve vacuum drying and molecular flow.

#### 5:00pm VT-TuA-9 Novel Cylindrical Hot Cathode Ionisation Gauge, *Ricardo A.S. Silva*, *N. Bundaleski*, *O. Teodoro*, CeFiTec - Nova School of Science and Technology, Portugal

Ionisation vacuum gauges are unexpendable devices for pressure measurement in HV, and particularly UHV and XHV. However, most of their commercial realizations (e.g. Bayard Alpert gauge) are known for their lack of accuracy, mainly due to the lack of electrodes robustness and the changes in the contributions of unwanted phenomena that occur during the operation. Among the latter , the most critical are photoelectron and ion induced electron emission from an ion collector electron stimulated desorption of ions and neutrals, as well as electron backscattering from an anode [1 3]. In the present work we report a design and realization of a new hot cathode ionisation gauge aiming the suppression of these unwanted phenomena in order to obtain increased accuracy, stability and low pressure limit. In this gauge , the primary electrons form a belt like beam, following curvilinear paths in an electrode assembly resembling a cylindrical energy analyser, and end their trajectories in a Faraday cup, located inside the inner cylinder electrode [4]. The ions created by electron impact with the gas are accelerated radially towards the ion collector, practically representing one of the electrodes of the "cylindrical energy analyser". The simulations of the operation, based on a recently developed approach [5], the construction details and the first experimental tests carried out with the first and second prototypes are presented. Details of the choice of the geometry of the Faraday cup and the inclusion of a suppressor electrode on the second prototype to inhibit secondary electron emission from the ion collector are also discussed.

#### References:

[1] K. Jousten, F. Boineau, N. Bundaleski, C. Illgen, J. Šetina, O.M.N.D. Teodoro, M. Vičar, M. Wüest, A review on hot cathode ionisation gauges with focus on a suitable design for measurement accuracy and stability, Vacuum 179 (2020) 109545

[2] H. Yoshida, K. Arai, Quantitative measurements of various gases in high and ultrahigh vacuum, J. Vac. Sci. Technol. A 36 (2018) 031604

[3] I. Figueiredo, N. Bundaleski, O.M.N.D. Teodoro, K. Jousten, C. Illgen, Influence of ion induced secondary electron emission on the stability of ionisation vacuum gauges, Vacuum 184 (2021) 109907

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[4] B. Jenninger et al., Development of a design for an ionisation vacuum gauge suitable as a reference standard, Vacuum 183 (2021) 109884

[5] R. Silva, N. Bundaleski, A. L. Fonseca, and O. M. N. D. Teodoro, 3DSimulation of a Bayard Alpert ionisation gauge using SIMION program,<br/>Vacuum,Vacuum,164(2019)300300

## 5:20pm VT-TuA-10 High Performance Sealing In Extreme Environments, *Christopher Cosgrove*, Technetics Group

This presentation details various methods of sealing to very low leak rates in extreme environments. These environments could be very high temperatures or cryogenic temperatures or very high pressures down to UHV.

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