

Vacuum Technology

Room 121 - Session VT2-MoA

Measurement, Partial Pressure, and Gas Analysis

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

2:00pm VT2-MoA-3 Monitoring Chamber Health with an Optical Plasma Gauge, *Martin Wüest, S. Kaiser*, INFICON AG, Liechtenstein

Leak testing is a common task in the daily laboratory routine. There are simple but lengthy procedures available to test for leaks, for example the rate-of-rise method. To check if a gas such as water or oxygen is below a certain concentration is often done just by waiting for a time that has been determined by experience. More powerful methods are also available such as mass spectrometers, in particular residual gas analyzers, or dedicated leak detectors. However, they tend to be expensive, are often not very easy to operate, and operate at low pressures. Alternative methods exist such as optical emission spectrometer. They tend to be bulky and extracting robust information is not that easy and below a certain pressure there is not enough light available to analyze.

We have now developed a compact optical plasma gauge to address the questions and shortcomings mentioned above. It combines a gas type monitoring optical plasma sensor with a total pressure sensor. Its design is optimized to allow a gas detection measurement in the range between 10^{-7} and 5 hPa. The gauge allows for the detection of gases such as oxygen, nitrogen, hydrogen or argon in-situ or in a rate of rise leak testing. Above 20 hPa the plasma generation is switched off in order to prevent plasma damage in the sensor. The total pressure sensor operates from 10^{-5} Pa to atmosphere. Discharge pressure is known to play a substantial role in the various competing collisional excitation and de-excitation processes that occur in the plasma. The measured optical spectrum is convoluted with the independent total pressure data to provide higher accuracy. The intelligence implemented directly on the gauge automatically configures the optimal measurement setting in order to ensure easy integration and optimized signal-to-noise ratio. Impurities > 10 ppm can be detected.

2:15pm VT2-MoA-4 Design and Construction of a Fixed Length Optical Cavity (FLOC) Pressure Calibration Standard for Calibration of Military and Commercial Aircraft, *Jacob Ricker, K. Douglass, J. Hendricks, T. Bui*, NIST

NIST has constructed several Fixed Length Optical Cavity (FLOC) pressure standards based on gas refractivity and shown that they are effective at measuring absolute pressure [1]. The US Air Force has recently funded development of these standards for the support of their Air Data Calibration Systems. These Air Data Systems provide calibration for altimeters and air speed indicators and traceability of these sensors is crucial for all operational military and commercial aircraft. The current US airspace requirements dictate every aircraft be calibrated at least every 2 years with a device that has an accuracy of pressure reading around 0.03% at pressures around $1/10^{\text{th}}$ of atmosphere.

The air force maintains hundreds of portable standards, working standards, and secondary standards worldwide to achieve that goal. Additionally, the Air Force also maintains other high-pressure standards to meet the operational requirements to provide calibration of pressures up to 10,000 kPa. The air force desires a high accuracy, portable standard that operates over the full pressure range using direct traceability via gas refractivity. A portable standard that is based on fundamental constants rather than frequent recalibration can be forward deployed and will save significant time and money for all civilian and military aircraft operators. With a redesigned FLOC, NIST believes it can meet all the requirements with one portable unit. This presentation will describe the design and construction of a new lower cost/robust/portable calibration system capable of calibrating gas pressure sensors over the entire range of 1 Pa to 10 MPa.

References:

[1] <https://doi.org/10.1016/j.measen.2021.100286>.

2:30pm VT2-MoA-5 Analysis and Adaptability of the ITER Diagnostic Residual Gas Analyzer Vacuum System, *Brendan Quinlan, C. Marcus, J. Perry, C. Smith III, C. Klepper, T. Biewer*, Oak Ridge National Laboratory

INVITED

The composition of exhaust gases in the divertor region is a critical measurement for long pulse devices like ITER. This measurement will provide important information for areas such as fuel-cycle processing and plasma heating [1]. The ITER Diagnostic Residual Gas Analyzer (DRGA) is

well suited to make these measurements because it is a multi-sensor diagnostic system capable of resolving isotopic compositions of hydrogen and helium as well as other heavier elements and compounds [1]. The DRGA will sample a slip stream of gas from the cryogenic pump duct via a sampling pipe that is approximately 7 meters in length and 70-100 millimeters in diameter. At the sampling pipe entrance, an orifice is present which creates molecular flow conditions in the entire length of the sample pipe. The sampling pipe has recently been updated to reflect necessary changes in the ITER port cell area. To provide measurements on timescales that are relevant, the DRGA vacuum system conductance must be revisited from [2] to ensure the appropriate response time and pressure can still be achieved. In this work, Molflow+, which is a Test Particle Monte Carlo (TPMC) simulation code, is used to simulate the conductance and assess the impact of the revised pipe routing. In addition to the new pipe routing, a new pumping scheme has been proposed in previous work and will provide the ability for variable pump speed [3, 4], while overcoming limitations of using an inter-stage port for the optical gas analysis [5]. In the event the orifice is restricted over time, the variable pump speed provides the added benefit of adaptability to ensure the appropriate conductance can be maintained. The restricted orifice is simulated using Molflow+ and compared to test data collected from an in-lab prototype. This study provides important guidance for the design of the ITER DRGA and confirm key operational parameters.

[1] C.C. Klepper et al., 2022 *IEEE TPS*, 50 (12) 4970-4979

[2] C.C. Klepper et al., 2021 *Fusion Science and Technology*, DOI 10.1080/15361055.2021.1898867

[3] C. Marcus et al., 2024 SVC TechCon, Pending

[4] B.R. Quinlan et al., 2024 *IEEE TPS*, DOI 10.1109/TPS.2024.3387443

[5] C.C. Klepper et al., 2017 *JINST* 12 C10012

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The views and opinions expressed herein do not necessarily reflect those of the ITER Organization.

3:00pm VT2-MoA-7 Effect of Thermal Transpiration on Calibration of Sapphire-Based Capacitance Manometer, *Kimihiko Sato*, Azbil Corporation, Japan

In semiconductor manufacturing, capacitance manometers are generally used for measuring the pressure during deposition or etching. These process gases are highly reactive and often corrosive and the manometers are often heated to 100 to 300 °C to prevent byproduct depositions inside them. Therefore, they are required to have high corrosion resistance and operate at these high temperatures. In order to meet these requirements, we have developed capacitance manometers equipped with a MEMS (Micro-Electro-Mechanical Systems) sensor chip based on sapphire, which has the excellent chemical corrosion resistance and thermostability [1].

Since manometers are often used at high temperatures, they must be calibrated at similar temperature at production. These products are heated to various temperatures depending on the target process, while the reference gauge is kept at a constant temperature (near room temperature) for the production efficiency. In this case, the pressure may be different between the product side and the reference side by thermal transpiration. It occurs when two vessels with different temperatures are connected by a narrow tube and the gas flow is in molecular or intermediate flow regime. Therefore, the difference must be compensated in the calibration process and we correct it based on the formula [2,3].

Conventionally, the instrumental error between the product and the reference gauge had been a typical specification (accuracy) that indicates the performance. In addition, the ISO standard [3] requires that the uncertainty be considered. It improves reliability of measurements and brings benefits to the users. In order to comply the standard, it is essential to consider thermal transpiration which is one of the uncertainty factors.

Since the uncertainty of the formula [2,3] is unknown, we studied the effect in the production facility by comparing experimental and simulated results. In the experiment, the pressure difference between the product side and the reference gauge side was measured. In the simulation, the temperature distribution of each part in the environment was obtained by thermal analysis, and the pressure distribution was obtained by Monte Carlo direct simulation (DSMC-Direct Simulation Monte Carlo). We show the results and consideration of the two efforts.

Monday Afternoon, November 4, 2024

[1] T. Ishihara, Upgrading a sapphire-based capacitance manometer for reduced size and enhanced anti-deposition characteristics, *azbil Technical Review*, April 2023.

[2] T. Takaisi and Y. Sensui: *Trans. Faraday Soc.*, 59 (1963) 2503.

[3] ISO 20146:2019, Vacuum technology — Vacuum gauges — Specifications, calibration and measurement uncertainties for capacitance diaphragm gauges.

3:15pm **VT2-MoA-8 Measurements of Electrode Temperatures in the Standardized Ion Reference Gauge, *Janez Setina***, Institute of Metals and Technology, Slovenia

A consortium of European National metrology institutes and industrial partners has recently developed a new type of reference ionization vacuum gauge. It is distinguished by its well-known sensitivity and excellent temporal stability, which is the result of straight electron trajectories. Electrons flying through the ionization volume have practically the same path lengths, so the probability of ionization of gas molecules is almost the same for all electrons [1]. The novel Ion Reference Gauge is on its way to standardization of electrode system configuration in ISO TS 6737.

The source of electrons in the gauge is the thermionic cathode, which heats the surrounding surfaces with thermal radiation. Therefore, the ionization cell has a higher temperature than the temperature of the vacuum system in which we want to accurately measure the gas pressure. In non-isothermal systems at low pressures (in the molecular regime), due to the so-called phenomenon of thermal transpiration, the pressure in parts with different temperatures is not the same.

In our research, we measured the temperatures of individual electrodes in the vicinity of the thermionic cathode with the aim of evaluating the influence of the phenomenon of thermal transpiration on the sensitivity of the gauge. In this talk, we will present the experimental setup and the obtained results.

[1] Jousten K, et al, Electrons on a straight path: A novel ionisation vacuum gauge suitable as reference standard, *Vacuum* 189, (2021), 110239

3:30pm **VT2-MoA-9 Calibrations of Spinning Rotor Gauges Towards International Comparison of Vacuum Standards, *Yoshinori Takei, H. Yoshida***, AIST, Japan

With the advancement of the semiconductor industry and the transition to a hydrogen energy society, the demand for vacuum measurement has surged. In such circumstances, the spinning rotor gauges (SRG) has garnered attention as one of the most accurate vacuum gauges capable of measuring the vacuum pressure range from 0.1 mPa to 1 Pa. In metrology field, the SRGs have been used as a reference standard for many years.

In national metrology institutes around the world, several vacuum standards such as static expansion system, optical pressure standard and orifice-flow method system are managed for the calibration of vacuum gauges like SRGs, diaphragm gauges, and ion gauges. These standards are based on physical principles, and their uncertainties are evaluated in each metrology institute. For instance, the static expansion system in Japan calibrates SRGs with a relative expansion uncertainty of 0.28% ($k=2$) [1]. Furthermore, this uncertainty is expected to improve further by combining static expansion system with the optical pressure standard [2]. To verify the consistency of uncertainty, national metrology institutes compare their vacuum standards. Since transporting the vacuum standards themselves for direct comparison is challenging, the same vacuum gauge (SRG) is transported. The calibration results for the same gauge with the vacuum standard of each country are compared. However, the reproducibility of SRG poses a challenge in this process. While SRGs are excellent vacuum gauges, their values may change by about 1 % during transportation, limiting the comparison accuracy of vacuum standards. Therefore, for enhancing the accuracy of international comparison of vacuum standards in the future, careful selection of superior SRGs and in-depth understanding of calibration conditions are essential.

Until recently, only one manufacturer produced SRGs. However, another company has recently entered the market, manufacturing and selling SRGs. There are some differences in the specifications of these SRGs. In this study, we experimentally confirmed the differences when using the SRGs for metrological purposes. Additionally, with the recent publication of ISO 24477 concerning the calibration of SRGs, which describes two calibration methods, we experimentally confirmed the differences. We also conducted calibrations by varying conditions such as the rotation frequency of the rotor, the rotor itself, and the mounting angle of the flange. These experimental results are shown in this presentation.

[1] Yoshinori Takei et.al., *Vacuum*, 187, 110034, (2021).

[2] Yoshinori Takei et.al., *Measurement: Sensors*, 22, 100371, (2022).

4:00pm **VT2-MoA-11 Vacrysim - Modeling Noise from Residual Gas for Cryogenic Interferometry, *Henk Jan Bulten***, Nikhef, Netherlands; *V. Erends*, High Voltage Engineering Europa, Netherlands; *B. Munneke*, Nikhef, Netherlands

INVITED

Since 2015, gravitational waves (small ripples in the fabric of spacetime) that arose from the mergers of black holes and/or neutron stars have been measured with the large, ultra-precise interferometers of LIGO and Virgo. Einstein Telescope (Fig. 1) is a plan for a next-generation gravitational-wave observatory with a strain sensitivity of 10^{-25} , allowing for precision tests of general relativity, cosmology and astrophysics. In order to reduce thermal noise, Einstein Telescope will operate with mirrors at cryogenic temperatures. This poses new stringent criteria on the vacuum system. Residual gas introduces noise via optical path length changes in the arms, Brownian motion of the mirrors, and ice build-up on the mirror coatings. Einstein Telescope requires residual gas pressures of below 10^{-10} hPa in the (10-km long) arms, and better around the cryogenic mirrors.

The design of the Einstein telescope requires accurate modeling of migration of molecules through the vacuum system, which contains complicated metal support structures and thermal shields, electronic devices with polymer cable mantles, silicon mirrors, etc. We want to model the outgassing and incident particle rate of all components as a function of time. This outgassing is strongly dependent on the history and on the applied temperatures.

Generally, heat flow and molecular flow predictions in finite-element based toolkits like Molflow or Comsol are calculated for steady-state; adsorption/desorption from the surfaces is taken as constant. However, to numerically calculate the time evolution of the system and see where the molecules deposit/evaporate one needs millions of time steps in which the coupled equations are solved. We developed a simulation toolkit, vacrysim, that is capable of this feat thanks to separating the time-independent tracking information from the time-dependent material properties information. Vacrysim can track thermal radiation and molecular paths and subsequently solve for conductive and radiative heat flow, and adsorption/desorption. Using vacrysim one can model the performance of cryogenic designs in terms of cool-down times, ice build-up and pump-down times. For instance one can predict the ice build-up on the mirror, and the time-dependence of the water distribution in a kapton-coated cable, after venting and evacuating a part of the vacuum system.

In this presentation we will address vacuum requirements for cryogenic interferometry and discuss the vacrysim toolkit.

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