Vacuum Technology
Room 104C - Session VT-TuM

Vacuum Pumping and Material Outgassing

Moderators: Martin Wüest, INFICON Ltd., Liechtenstein, Jacob Ricker, NIST

8:00am **VT-TuM1 Applicative Challenges for today's Turbo Molecular Pumps***, **Adrian Wirth**, H. Bernhardt,* Pfeiffer Vacuum GmbH, Germany*; N. Cotton,* Pfeiffer Vacuum Inc **INVITED**

Since its invention in 1958 by Mr. W. Becker, the turbo molecular pump (TMP) has been a milestone in showing new and effective ways of providing oil-free high vacuum. It soon started to replace available pumping principles and as the need for high vacuum grew, it became the standard for modern high vacuum applications. It has since been confronted with increasingly diverse customer needs and performance challenges.

In most aspects, the requirements can be subdivided into primary (pump performance related) properties like pumping speed, gas throughput, compression, fore-vacuum compatibility and secondary ones. The latter including attributes of compactness, ease of system integration, corrosion and condensation insensitivity, usability in areas exposed to ionizing radiation, maintenance friendliness, outgassing and particle cleanliness, emission levels of vibrations, sound or electromagnetic radiation, lifetime and costs of ownership—just to name a few.

From the beginning, demanding applications and processes were on the one hand influencing the vacuum performance of the TMP, hence indirectly affecting the effective shape of the rotor and stator parts and its combination with other pumping principles. On the other hand, triggered by specific and extreme applications or operation settings and surroundings, the secondary requirements gained in importance.

Within this scope, some of today's applicative challenges and customer needs for TMPs will be presented. Examples will include the operation of TMPs exposed to magnetic fields in ion implanters or ionizing radiation at particle accelerators, in magnetic stray field and vibration sensitive systems such as electron microscopes, integrated into systems in analytics where low sound levels and the ease of integration are of special interest as well as in corrosive processes. Multi-port characteristic applications with rotor designs being specifically tailored for the individual vacuum performance require sophisticated calculation know-how. Apart from these operation purposes, the utilization in tool coating, nuclear fusion experiments, XUV lithography and mobile employments impose particular specifications. Furthermore an overview and comparison of process/requirement specific TMP designs will be provided.

The TMP had and still has to meet most diverse applications and hence undergoes a change from purely vacuum performance driven specification inputs into a period where also secondary requirements are ever since gaining in importance and hence impose a continuous evolution of the TMP technology.

8:40am **VT-TuM3 Ion Pump Design for Improved Pumping Speed at Low Pressure***, **Alessandro Abatecola**, M. Audi,* Agilent Technologies, Italy

Although Ion pumps are widely and mostly used in UHV conditions, virtually every existing Ion Pump has its maximum pumping speed around 10 E-6 mbar (10E-4 Pa). The discharge intensity in the Ion Pump Penning cell (the number of ions that bombard the cathode per unit time) is pressure dependent, and it is the main parameter that influences the pumping speed.

A study has been performed to evaluate the influence of magnetic fields and cell dimensions on the Ion Pump Discharge Intensity at different pressure. As a result, a combination of parameters has been defined that allows the design and manufacture of an Ion Pump with maximum pumping speed shifted towards lower pressures.

Experimental results with several different experimental set ups are presented.

A new 200 L/S Ion Pump specifically designed for UHV operation that incorporates these findings to obtain maximum pumping speed in the 10E-8 mbar (10E-6 Pa), and with all components subject to Vacuum Firing prior to assembly to obtain the lowest ultimate pressure is described.

9:00am **VT-TuM4 Multi Scaled Titanium Gettered Surfaces for Enhanced Pumping of H_2***, **Alan Van Drie**,* Tri Alpha Energy

The sticking factor for any gas onto a flat surface, whether its physisorbed or chemisorbed can be enhanced by increasing the effective roughness of the flat surface. When done on multiple scale lengths, not unlike a fractal pattern, the sticking factor can approach unity. In the present case, we are interested in pumping H_2 by a fresh coating of titanium, which has a sticking factor of around 3-6% at room temperature on a flat surface. The shape of the fractal pattern and number of scale lengths that can be used is limited by the feasibility of coating titanium on all the exposed surfaces. Using three distinct scale length from ~20 cm, ~2 cm to sub-micron we are able to obtain an estimated 70-80% effective sticking factor. The sub-micron scale is achieved by depositing the titanium onto a cryogenically cooled surface that changes the film's morphology from a dense coating to a porous sub-micron needle like structure. This by itself increases the sticking factor from the normal 3-5% to 20-50%. Some deposition techniques can even create film morphologies with up to 90% sticking factor; eliminating the need for other macroscopic scale lengths. Using these techniques we can create a 2,000 m^3/s pump for H_2, with 10 m^2 entrance area that fits within our 15 m^3 vessels. The design principles and preliminary pumping speed and capacity measurements will be presented.

9:40am **VT-TuM6 Outgassing of UHV Stainless Steel Cans***, **Lily Wang**, P.D. Honnell,* Los Alamos National Laboratory

UHV stainless steel cans are used to contain material samples in our experiments to study how the materials age under accelerated thermal conditions. In addition to characterizing the material properties after the thermal aging treatment, we also measure the amount of gas released during these thermal aging runs and analyze the off gas collected. In preparing for the experiments, the stainless steel cans were first vacuum-baked at 150°C to remove adsorbed moisture. The vacuum-baked cans were then brought into a dry nitrogen glovebox where the samples were loaded into these cans. The experiments were conducted in static vacuum at various temperatures ranging from 25 to 145°C for a duration ranging from 25 days to several years. The pressure of the gas accumulated in the sample can was measured with a capacitance diaphragm gauge. At each temperature, an empty UHV stainless steel can was also measured. Stainless steel contains dissolved hydrogen that diffuses out and contributes background outgas in vacuum systems. This talk will present the gas evolution results obtained from these empty cans and discuss how this stainless steel outgassing affects our gas evolution measurements.

11:00am **VT-TuM10 Calibration of Reference Samples for Water Vapor Outgassing and Water Vapour Transfer Rate***, **Janez Setina**,* Institute of Metals and Technology (IMT), Slovenia*; K. Jousten,* Physikalisch-Technische Bundesanstalt (PTB), Germany

Water vapor is usually main constituent of residual gas in non-baked high vacuum systems. The principal source of water vapor is outgassing from surfaces of vacuum chamber. Significant amount of water vapor is adsorbed on the surfaces every time when the system is vented to moist ambient air. Outgassing rate of water vapor is therefore important parameter of any vacuum material and is needed for proper design of the system to operate in required pressure range.

Traceability of outgassing rate measurements was one of the research activities of the recently finished European project EMRP IND12. For calibration, validation and comparisons of outgassing rate measurement systems different reference outgassing samples were studied. In this presentation we will focus on the reference samples for water vapor outgassing, which were jointly developed and applied for patent by PTB and IMT [1]. For measurement and calibration of outgassing rate from reference samples for different gases, including water vapor, a dedicated vacuum system was developed at IMT. Measurement range of the system for water vapor outgassing rate at room temperature is from 5×10^{-7} mbarL/s to 5×10^{-3} mbarL/s (equivalent to 2×10^{-11} mol/s to 2×10^{-7} mol/s, or 3×10^{-5} g/day to 0.3g/day). Typical relative uncertainty of measured outgassing rate is below 5 %.

Results of calibration of two water vapor reference samples will be presented. One sample had nominal outgassing rate of water vapor 6×10^{-7} mbarL/s (2.5×10^{-11} mol/s, or 4×10^{-5} g/day) and another sample 3×10^{-5} mbarL/s (1.3×10^{-9} mol/s, or 2×10^{-3} g/day). Both samples represent constant outgassing rate over long period of time. Repeated measurements showed time stability of reference samples better than 3%/year.

The newly developed water vapor outgassing reference samples can be also used for calibration of measurement instruments for water vapor transfer rate (WVTR).

[1] Patent DE 102014200907 A1 (2015)

11:20am **VT-TuM11 Simulated and Measured Extreme High Vacuum in the Jefferson Lab Polarized Electron Source***, **Marcy Stutzman**,* Thomas Jefferson National Accelerator Facility

The polarized electron source for the Jefferson Lab CEBAF nuclear physics program has stringent vacuum requirements for successful operation. Research projects aimed at improving the static vacuum into the extreme high vacuum range, below 10^{-12} Torr, have investigated outgassing rate reduction through coatings and heat treatments, vacuum characterization optimizing the utilization of commercial extreme high vacuum gauges, and pumping configurations including developments in UHV/XHV cryopumping. Additionally, limitation of dynamic vacuum during operation has been studied using surface analysis and processing toward reducing field emission from the high voltage electrode in the electron source. Both modeling and experimental results of these studies and the impact of incorporating these improvements in the vacuum system for the Jefferson Lab polarized electron source will be presented.

11:40am **VT-TuM12 Outgassing Rate Measurements of 3-D Printed Materials***, **Makfir Sefa**, J.A. Fedchak, J. Scherschligt,* National Institute of Standards and Technology (NIST)

3-D printing of parts has many potential advantages over traditional machining. The outgassing rate of these materials is particularly interesting for determining their performance in a vacuum environment and for other practical applications. We measured outgassing rate of 3-D printed stainless steel and titanium samples. The outgassing rate was measured using pressure rate-of-rise method and throughput method. The outgassing rate of each sample was measured at room temperature. The composition of the desorbed gas was also determined. The experimental results and measurement procedure will be discussed in the presentation.

12:00pm **VT-TuM13 Characterization Studies of UHV Polished Surfaces***, **Melisa Buie**, C. Fields,* Coherent Inc*; A. Cress,* San Jose State University

Interest continues to remain high in the application of electropolished surfaces. Studies of outgassing and surface treatments dating back to 1969 have remained relevant as science continues to push the lower boundaries of UHV. [1-5] More recently, polished stainless steel surfaces have been tested for use in high voltage applications. [6] The stainless steel flanges must have excellent sealing capability in low compression UHV joints, be able to withstand temperatures above 1200°C, increased corrosion resistance, and provide minimal outgassing.

A characterization study was performed comparing electropolished stainless steel flanges with plated and subsequently hand-polished stainless steel flanges to optimize the flange surface for temperature resistance, corrosion resistance, hermeticity, and outgassing. A randomized designed orthogonal experiment (full factorial) was performed varying the type of polish, the electrolytic exposure time, and post-polish processing temperature. Electroplished surface quality showed a strong interaction between the exposure time to the electrolytic bath and the post-process annealing temperature. Nickel electroplating followed by hand polishing showed resultant plating thickness inconsistencies along with an adverse effect at elevated temperatures.

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[3] K. Odaka and S. Ueda, J. Vac. Sci. Technol. A 13, 520 (1995).

[4] Y. Sasaki, J. Vac. Sci. Technol. A 25, 1309 (2007).

[5] P. Nunez, E. Garcia-Plaza, A. R. Martin, R. Trujillo and C. Dela Cruz, AIP Conf. Proc. 1181, 130 (2009).

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