

Tuesday Evening Poster Sessions, October 22, 2019

Vacuum Technology Division Room Union Station AB - Session VT-TuP

Vacuum Technology Poster Session

VT-TuP-1 Dynamic High Pressure Technique for Surface Analysis of Gas Sensors in Quasi-operating Condition, Taku Suzuki, Y Adachi, I Sakaguchi, National Institute for Materials Science (NIMS), Japan

One of the biggest problems in surface analysis of gas sensors is the pressure gap; a conventional surface analytical tool needs high vacuum (10^{-4} Pa), while gas sensors are usually employed in atmospheric pressure (10^5 Pa). This problem has been partially overcome by recent operando techniques for surface characterization. In such a operando measurements, either effective differential pumping or a pressure separation technique, which is often called a high pressure cell, are typically utilized. With those techniques, surface analytical tool can be operated in (ultra-) high vacuum while keeping the pressure in the vicinity of a sample (near) atmospheric pressure. Those techniques are obviously useful to analyze gas sensing mechanism on surfaces. However, a high-speed evacuation system is expensive and a membrane for separating pressure limits applicable analytical techniques.

Besides above-mentioned operando measurements, a dynamic high pressure (DHP) technique has been proposed to analyze a device surface in operating condition. Briefly, it is a technique of pulsed-gas injection to a sample surface. The technique seems attractive considering economical cost and possible wide range of applicability. However, the number of reports concerning DHP is quite limited, and thus, it is not clear whether DHP is useful for surface analysis of gas sensors.

In the present study, we have developed a pulsed gas injection system of pure air combined with an ultra-high vacuum chamber and a fast pressure transducer. The pressure at the sample position reached at about 10^3 Pa and 10^4 Pa with pulse width of 10 ms and 100 ms, respectively, with the inlet pressure of 1 MPa. The background pressure was below 10^{-2} Pa with pulse width of 10 ms except for the duration of 1 s after the gas injection. We further developed a gas sensor measurement system combined with the pulsed gas injection system. In our preliminary experiment using a W-ZnO thin film gas sensor, we successfully observed substantial change of electric resistance with introducing the pulsed pure air by using a lock-in technique.

VT-TuP-2 Fundamental Study for Practical Applications of Ti-Zr-V NEG Coating to General Vacuum Systems, Makoto Okano, A Niwata, S Kitamura, JEOL Ltd., Japan; Y Tanimoto, X Jin, M Yamamoto, T Honda, High Energy Accelerator Research Organization (KEK), Tsukuba, Japan

Modifying the properties of surfaces has become essential to obtain a desired function in various ultra high vacuum (UHV) systems. Among such techniques, Ti-Zr-V non-evaporable getter (NEG) coating, originally developed at CERN^{1,2)} and being widely applied to particle accelerators, is one of the most promising functional coatings, as it provides high effective pumping speeds, low outgassing rates, and low secondary electron yields. Since these desirable properties are beneficial in any UHV systems, there has been an increasing demand for its widespread availability. Furthermore, NEG coating is expected to maintain UHV conditions in power-less situations; for example, its application to electron microscopes might enable long-sustained transportation and quick recovery to UHV. For these practical applications to general vacuum systems, we have started a fundamental study on NEG coatings, where the vacuum properties are measured by a build-up method and the durability of the pumping capacity is examined by repetitive cycles of air-exposure and activation. In order to establish a technique to deposit high-performance films on various vacuum chambers by magnetron sputtering, the coated surfaces are characterized by scanning electron microscope (SEM), energy dispersive X-ray spectrometry (EDS), and X-ray diffraction (XRD). The test tubes used in the build-up experiment are made of 304 stainless steel and measures 50 mm in diameter and 300 mm in length. One tube is coated with 0.7 μ m Ti-Zr-V films and the other is uncoated. After 24 hours of stopping the sputter ion pump (SIP), the pressure in the uncoated tube increased from 2E-8Pa to 3E-6Pa, while the increase was suppressed from 6E-9 Pa to 1E-8Pa in the coated tube. Even after an additional build-up for 10 days, the coated tube was maintained under UHV conditions (7E-6Pa), and the pressure was recovered to 5E-8Pa in 5 hours after switching on the SIP. A comparison by residual gas analysis after the 24-hour build-up showed that the NEG coating improved 360-times for CO and 100-times for H₂. These results

suggest a feasibility of the transportation of UHV systems without electricity. The presentation will include preliminary results of film characterization by the surface analyses, as well as pumping properties of the NEG coating.

References

- 1) C. Benvenuti et al., Vacuum 60 (2001) 57.
- 2) P. Chiggiato and P. Costa Pinto, Thin Solid Films 515 (2006) 382.

VT-TuP-3 Fabrication and Characterization of a Variable Conductance Vacuum Valve to Control Pressure Level for a High Vacuum System, Han Wook Song, S Woo, Korea Research Institute of Standards and Science, Republic of Korea

Fabrication of a semiconductor device requires a high precision and a high degree of cleanliness. For this reason, the semiconductor device is manufactured in a state in which the contact of the foreign substances contained in the air is completely blocked, that is, in a vacuum state (approximately around 0.1 Pa.) To adjust the required vacuum level, some apparatus such as a needle valve, a gate valve, and a butterfly valve were used to control the gas flow. Recently, we developed a variable-conductance vacuum valve to control the inlet pressure for vacuum chamber, which was modified from optical iris used in optics. The present conductance variable valve is characterized in that the conductive tuck adjusting portion and the conductance operating portion employing the iris structure do not have a physically contacted or coupled structure. And the housing has no mounting holes, fastening holes, or the like for coupling the conductance operating portions, so it has a conductance variable function that can completely block the fluid leakage of the valve without any separate parts such as packing, sealing, and O-ring. The guide section of the housing will have a first magnetic body, while the conductance control unit will have a second magnetic body in the mounting hole. Therefore, when the first body and the second body are moved in a circular direction by the attraction force, the first body within the guide unit also moves in a circular direction. The cross sectional area of housing and the conductance of fluid are proportional. When using the manufactured variable valve, it showed 2% reproducibility and 0.5% repeatability in pressure generation. In the future, we will make and evaluate a large variable valve that can be applied to high vacuum systems.

VT-TuP-4 Hellum Gas Transmission Rate of Elastomer Seal with a Back-up Ring Seal, Masaharu Miki, EM Technical Lab Inc., Japan; S Nowatari, H Hanada, IIDA Co., Ltd, Japan

Hellum gas transmission rate of elastomer seal with a back-up ring seal was studied using the metal-sealed Hellum leak detector. Three kinds of samples were prepared. One is an elastomer seal (JIS B 2401 V40) without a back-up ring seal. Second is the same size elastomer seal with a back-up ring seal which adheres to the atmospheric-side surface of the elastomer seal and is made of some resin. The last is the same one with a back-up ring seal except the seal surface of the back-up ring seal is not flat but having some structure. Hellum gas transmission rate was measured and evaluated. It was found that the elastomer seal with the back-up ring seal has very low Hellum gas transmission rate, which is about less than 10% of the case without a back-up ring seal. It is like the case of a metal seal. On the other hand, the difference of the seal surface of the back-up ring seals was not found. Anyway, the elastomer seal with the back-up ring seal must be useful as seals to make vacuum chambers up-grade which structure cannot permit to use any metal seals. Pumping-down curves on total vacuum pressure and residual gas pressure (O₂, H₂O, etc.) in a vacuum chamber using the elastomer seal with the back-up ring seal are under investigation.

VT-TuP-5 Improved NEG Sputter Deposition System, Philip Adderley, M Stutzman, Jefferson Lab

Jefferson Lab been using a DC sputtering system to coat the beampipe with a Ti-Zr-V non-evaporable getter coating for 20 years. A similar system has also been used to sputter coat large diameter chambers with ultimate pressures approaching XHV(1). The approaching upgrade of the Jefferson Lab CEBAF injector will use NEG coated beampipes of varied diameters for the first 30 meters of the machine. We describe the improvements that are being made to the sputtering system to improve stability, increase monitoring capabilities, and improve NEG film adhesion and morphology.

- 1) M.L. Stutzman, P.A. Adderley, A.A. Mamun and M. Poelker, J. Vac. Sci. Technol. A 36 031603 (2018).

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VT-TuP-6 Concepts for Reduced Load UHV Sealing Applications, *Ryan McCall*, Technetics Group

Designing a robust vacuum connection that can deliver UHV or XHV leak rates, withstand high temperature bake-outs and minimize hardware/bolting size can be difficult to achieve. The design process is complicated even further with Aluminum flanges or when a large diameter or shaped seal is required.

This presentation will review UHV metal sealing concepts and will detail a seal option that significantly reduces seating load by concentrating contact stress to a small area machined into the seal surface. This combination of load concentration and material selection allows for a helium tight seal with much less load than a traditional metal seal without damaging the corresponding flanges and hardware. The resilient seal loading mechanism also allows the seal to perform well in temperature cycles or bake-outs.

VT-TuP-7 Quantitative Gas Analysis with Quadrupole Mass Spectrometers - Comparison and Limitations, *Gregory Thier, L Kephart*, Extrel CMS; *T Whitmore*, Henniker Scientific

There are many factors to consider when comparing the overall suitability of different quadrupole-based gas analyzers for a given application. These can be categorized into two main areas, inlet/interface suitability and quadrupole mass analyzer suitability.

The suitability of the quadrupole mass spectrometer determines very important figures of merit such as precision, stability and detection limit. The quadrupole mass spectrometer includes the ionization method, the transmission characteristics, and the quality of the driving electronics.

Unfortunately, these figures of merit are often misrepresented in the commercial literature and it's this confusion which we seek to address and clarify in this document by making a direct comparison between two different classes of quadrupole analyzers; a typical 6mm rod diameter RGA type instrument typical of many currently on the market, and a higher performance 19mm rod diameter quadrupole analyzer, typical of high end analytical analyzers used in research and industry. We compare these with nominally identical inlet/transfer conditions, so that only the mass spectrometer performance is under consideration. In doing so, we present a direct comparison as it relates the various figures of merit and attempt to remove some of the mystery surrounding confusing analyzer specifications so that potential users of this powerful analytical technique may query manufacturer specifications and therefore make more informed decisions.

The specifications that we will discuss are:

- Detection Limit (minimum and maximum detectable concentration)
- Speed of Analysis (measurement speed and response time)
- Analysis Precision (repeatability of measurements)
- Analysis Stability (long-term instrument stability)
- Dynamic Range (comparison of largest and smallest detectable signals)

We will study the above by assessing and comparing the performance of two instruments, the MAX300-CAT and the MAX300-LG. The MAX300-CAT is typical of the high-end RGA based gas analyzers, based upon 6mm quadrupole rod technology, whereas the MAX300-LG is a higher performing analyzer based on 19mm quadrupole rod technology and high-performance electronics.

VT-TuP-8 Recent Developments of Home-made UHV SPM Systems and their Applications, *Qing Huan, Z Wu, R Ma, G He, Z Gao, L Bao, J Yuan, K Jin, H Gao*, Institute of Physics CAS, China

Scanning probe microscope (SPM) is a powerful tool for studying physical and chemical processes at single molecular/atomic level. The first part of this report will introduce our R&D progresses on UHV-SPM systems, which includes the update of a 4-probe STM, Variable-temperature STM, Low-temperature SPM combined with MBE and optical accesses, and Low-temperature SPM combined with PLD et. al. The second part will mainly introduce some research works on graphene, organic functional molecules and so on which are carried out on these home-made systems.

VT-TuP-9 An Experimentally Backed Modeling of NEG Pump Operation During Saturation, *Derek Hammar*, Coe College; *Y Lushatak*, Cornell University

Non-Evaporable Getter (NEG) pumps are increasingly common in particle accelerator applications

because of their small size and their strong performance for hydrogen, the principal UHV gas.

However, these pumps present a challenge to vacuum system design because their complicated

geometry results in unreasonably complicated vacuum simulations. This project seeks to build 3D

models of NEG pumps and their environments in AutoDesk Inventor and simulate their performance

in MolFlow, creating a database to NEG performance under various installation geometries and

attempting to simplify the pump geometry without sacrificing simulation accuracy. Key results are

verified experimentally

VT-TuP-10 3D printed Mini-Channel Plates – Vacuum Compatibility and Detector Performance, *Maram Alnahhas, J Moore*, Robot Nose Corporation

Conventional production of microchannel plates (MCPs) produces linear channels. Our simulations promise a significant improvement in time resolution by changing the geometry of the channels from straight to a zig-zag (Z) configuration, which drives the electrons to land on a specific surface in each gain stage, yielding higher time resolution. The sensitivity is improved as well by controlling the outer shape of the pores; replacing the circular pore pattern with hexagon or square will increase the open area ratio for the same MCP diameter. 3D printing MCPs not only enables this Z channel approach, but is a cost-effective method to produce charged particle detectors.

3dMCPs were created using a 70-micron resolution stereolithography printer. Both linear and Z channels were printed with 30:1 length to diameter (l/d) ratio. Graphite and other dopants have been used to change the resistivity of the printed material. In principle, the 3dMCP can then be coated for secondary emissions using ALD technology. Optical microscopy of the 3dMCP and its cross-section show that good uniformity can be attained throughout once the process was optimized.

To facilitate low vacuum flow testing of the 3dMCPs, a microcontroller based multi-vacuum gauge has been constructed. This device fits on a 2.75" Conflat flange with a built-in pressure gauge and OLED display. The gauges chosen are a Pirani pressure sensor calibrated with a capacitance manometer. To digitize the signals and establish serial connections between the electrical parts, the Trinket M0 MCU was used with a built-in 12-bit ADC and a reference voltage of 3.3v. I have also designed an enclosure that could easily attach to a flange.

An apparatus was built to measure the gain and pulse output characteristics of the MCPs. TauZero (τ_0) consists of an ultrahigh vacuum chamber with 2kV electron and 20kV Ga⁺ guns directed at multiple test 3dMCPs. The turbo pumped vacuum system is vibrationally isolated. τ_0 's electronics suite includes a 6 ½ digit DMM, 16 GHz digital oscilloscope, spectrum analyzer, a time-to-digital converter with 12ps resolution, and a digitizer with pulse height analysis software.

One of the main concerns regarding 3d printed materials is whether the plastic used will contaminate a high vacuum system. 3dMCPs were outgassed in a vacuum chamber while monitoring with a residual gas analyzer to collect data during the outgassing process. Vacuum did not damage the 3dMCPs and a drop of volatiles over 24 hours was measured. We concluded that the 3dMCPs are high vacuum compatible.

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