

# Tuesday Evening Poster Sessions, October 23, 2018

## Vacuum Technology Division Room Hall B - Session VT-TuP

### Vacuum Technology Division - Poster Session

**VT-TuP-1 Characterization and Imaging of Surface Acoustic Waves on GaAs with Raman Spectroscopy**, *Brian Rummel*, University of New Mexico; *M Henry*, Sandia National Laboratory; *S Han*, University of New Mexico

Surface acoustic wave (SAW) devices are commonly found in sensors and RF filters, and utilizing a facile technique to image the transmitted signal would prove useful in characterizing device operation and optimization. We show how Raman spectroscopy can offer analytical insight into the mechanical strain imposed by SAWs traveling along the surface of various III-V substrates. SAWs are generated using a single port interdigital transducer (IDT) design, modified to produce free surface standing waves. These standing waves provide a means to differentiate between nodes and antinodes of the acoustic wave. The temporal period of the SAWs does not easily allow *in-situ*, real-time measurement of the waves; however, a broadening of the Raman peaks corresponds to an averaging of the peak shifts over the integration time of the spectrometer. An analytical fitting model has been derived to effectively calculate the maximum strain induced by the acoustic waves, thus allowing one to characterize the SAWs. IDTs were deposited onto a GaAs (110) substrate to study the potential of Raman analysis for SAW devices. Wavelengths ranging from 3.2  $\mu\text{m}$  to 10  $\mu\text{m}$  were used to study insertion loss, attenuation, diffraction parameters, and the mechanical coupling coefficient. Future applications of this technique to probe growth defects in ScAlN/Si substrates will also be discussed.

**VT-TuP-2 Sapphire MEMS based Capacitance Manometer for Vacuum Freeze-Drying Device**, *Masashi Sekine*, *M Soeda*, *T Ishihara*, *M Nagata*, Azbil Corporation, Japan

New capacitance manometer which has high durability against Clean in Place (CIP) and Sterilization in Place (SIP) cleaning processes for vacuum-freeze drying equipment have been developed.

Vacuum-freeze drying equipment used for manufacturing of medicines or fine chemicals, CIP and SIP processes must be executed to ensure the sterilization of inside of the equipment according to International Organization for Standardization (ISO). The first CIP process, equipment inside is cleaned by water spray at room temperature and then cleaning is followed by SIP process with water vapor of 200-300kPa. To ensure whole cleaning, devices attached to equipment cannot be removed from the equipment. Also protecting valves for devices are not allowed because that inside of devices must be cleaned and stabilized. Therefore devices for these application should have durability of these water spray or vapor environments. Generally pressure range of a manometer for vacuum-freeze drying process is 100Pa absolute and it is heated up to 125 degree C for sterilization.

Authors have developed entirely sapphire-based capacitive pressure sensor chips utilizing MEMS (Micro Electro Mechanical Systems) processes, which are operated at up to 200 degree C with from 0-13 to 0-133k Pa pressure range mainly for semiconductor manufacturing. Cross sectional view of sensor package is shown in Fig.1. Cross sectional view of sensor chip is shown in Fig.2. Deformation of sapphire diaphragm due to pressure change is measured as capacitance change between sensor diaphragm and base-plate.

In the process of adopting this manometer into vacuum-freeze drying process, zero point shifts were found after CIP and SIP processes. Detailed investigation revealed the mechanism of these zero shifts as follows.

Once CIP and SIP processes are conducted, sensing diaphragm is contacting onto base-plate due to over pressure load, such as atmospheric pressure or 200-300kPa. Then diaphragm is mechanically deformed due to temperature change caused by cleaning water or devolatilized vapor. Then diaphragm slides on base-plate to cause friction. Since sapphire is highly electrically insulating material, this friction results in charging up on contacted surfaces of diaphragm and base-plate (Fig.3).

To solve this problem, we have developed a new electrode configuration which discharges static electricity. Also, improved diaphragm and sensor package structure to suppress thermal deformation have been designed (Fig.4). In actual CIP and SIP processes with this improved sensor structure, the zero point shifts were reduced to under 0.1% Full Scale, which is sufficient for this application (Fig.5).

**VT-TuP-3 Development of Vacuum Equipment Trainer (VET) Systems for Off-site Students**, *Delmer Smith*, *N Louwagie*, Normandale Community College

While the shape of the Internet of Things (IoT) is still evolving for manufacturers and consumers, its arrival is not in doubt. In addition to a strategic research and development plan, businesses must have a workforce with the knowledge and skills to respond to new directions. Normandale Community College in Bloomington, Minnesota, offers a 12-credit Vacuum Technology Certificate that can be completed in a year. The Vacuum and Thin Film Technology program (Part of the Engineering Technology Department) has provided local education in vacuum science for over 20 years at Normandale Community College. Utilizing technology and curriculum developed with the support of the National Science Foundation division of Advanced Technological Education (DUE 1400408 and DUE 1700624), the college now has the capability to reach students at multiple locations throughout the United States. Courses are delivered locally or via a telepresence interface to students throughout the United States. Students receive hands-on practice with Vacuum Equipment Trainer (VET) systems. The VET systems are modular. As students progress through courses, capability is added to increase system functionality. Normandale Community College currently has eight copies of the VET systems. This means that students, both onsite and offsite, can complete a Vacuum Technology Certificate in a year. The first remote student certificates were awarded in the spring of 2018. This paper describes the VET system functionality and how it is used to support the technical content students learn in class.

**VT-TuP-4 Vacuum System of the SuperKEKB Main Ring in the Phase - 2 Commissioning**, *Yusuke Suetsugu*, *K Shibata*, *T Ishibashi*, *M Shirai*, *S Terui*, *K Kanazawa*, *H Hisamatsu*, KEK, Japan

The SuperKEKB is an electron-positron collider with asymmetric energies in KEK aiming a high luminosity of  $8 \times 10^{35} \text{ cm}^{-2} \text{ s}^{-1}$ . The main ring (MR) consists of two rings, that is, a positron ring with an energy of 4 GeV (Low Energy Ring, LER) and an electron ring with 7 GeV (High Energy Ring, HER). Both rings have the same circumference of 3016 m. In the Phase-1 commissioning from February to June 2016, the vacuum system of the MR worked well as a whole at stored beam currents of approximately 1 A. However, several problems were found, and various countermeasures were taken against these during a long shutdown period before starting the Phase-2 commissioning. For example, permanent magnets were placed around the beam pipes to suppress the electron cloud effect (ECE) in the LER. Other than these works, new beam pipes and components were installed in the main ring, such as six beam collimators required for reducing the background noise of the particle detector. The Phase-2 commissioning of the MR started in March and will be continued until July 2018. By the end of April, the total beam dose (integrated beam currents) are approximately 52 Ah and 57 Ah, and the maximum beam currents are 0.32 A and 0.26 A for the LER and HER, respectively. The pressures recovered soon after starting the commissioning to the level at the final stage of Phase-1 commissioning, and further decreased steadily. The pressure rise normalized by a unit beam current are approximately  $1 \times 10^{-6} \text{ Pa A}^{-1}$  and  $2 \times 10^{-7} \text{ Pa A}^{-1}$  for the LER and HER, respectively. The beam lifetime of the HER is approximately 500 min. at 0.22 A, and is almost determined by vacuum pressure. On the other hand, that of the LER, approximately 90 min. at 0.3 A, seems to be mainly limited by the Touschek effect rather than the vacuum pressure. The results of various countermeasures taken in the shutdown period and the effect of permanent magnets or solenoids against the ECE will be investigated soon. The performances of the new vacuum components will be also checked at higher beam currents. Reported here will be the status and the major results of the vacuum system of the MR during the Phase-2 commissioning, and problems for the next Phase-3 commissioning.

**VT-TuP-5 Smart Diagnostics for Dry Vacuum Pumps Running in Semiconductor Processes**, *Wan-Sup Cheung*, *J Lim*, KRISS, Republic of Korea; *N LEE*, *J LEE*, *T Park*, *T Kim*, SK Hynix, Republic of Korea

This paper addresses logical and smart ways of diagnosing the operation conditions of dry vacuum pumps in the semiconductor processes of the SK Hynix company. In the last four years, SK Hynix has been attempting to complete the 'production data logging' systems, including even the collection of the state variables of dry vacuum pumps running in the semiconductor processes. Recent attempts have been added to exploit the logged state variables to diagnose individual dry vacuum pumps operating in the highly tough and risky production processes since their failures always accompany much time and cost. Adaptive parametric modelling

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(APM) approaches developed by the KRISS research team were considered to analyze the trend and statistical properties of the logged state variables of vacuum pumps. APM approaches are illustrated to provide merits of dramatic (more than 100 times) data reduction for the construction of 'normal operation condition (NOC)' reference batches from the beginning part of the logged state variables. The singular value decomposition (SVD) is exploited to decompose the selected NOC reference batch matrix into the core and projection matrices, which are shown to enable the evaluation of two diagnostic indicators for each vacuum pump, the Hotelling's T-squared (T2) value and the sum of squared residuals (Q). Both scalar values T2 and Q are demonstrated to play critical roles in diagnosing the current operation conditions of individual vacuum pumps, more specifically the similarity and difference of the current process state in reference to the NOC reference batches. In this work, several attempts have been carried out to improve the reliability and robustness of the diagnosis of vacuum pumps. The use of vibration measurements was the first selected candidate. Field tests are now in progress. It is expected that field test results are to be illustrated in the presentation of this paper.

**VT-TuP-6 Commissioning of Vacuum System for Positron Damping Ring for SuperKEKB, Kyo Shibata, Y Suetsugu, T Ishibashi, M Shirai, S Terui, K Kanazawa, H Hisamatsu, KEK, Japan**

The SuperKEKB, which is an upgrade of the KEKB B-factory, is a high-luminosity electron-positron collider. To satisfy the requirements of high beam quality for positron injection into the SuperKEKB, a new damping ring (DR) with a circumference of 135.5 m was constructed in an upgraded injector system. The positron beam extracted from the injector linac with an energy of 1.1 GeV is stored in the DR for more than 40 ms, where the beam emittance is damped. Subsequently, the low-emittance beam is extracted and sent back to the linac to be accelerated to 4 GeV and injected into the SuperKEKB. The maximum stored beam current is 70.8 mA.

The vacuum system of the DR is divided into five sections. Main sections are two arc sections with a length of ~110 m in total. Since there is synchrotron radiation irradiation in the arc sections (critical energy: 0.8-0.9 keV), the average pressure over the entire ring during the beam operation is determined mainly by the pressure of the arc sections. The material of the beam pipes is aluminum alloy. The required beam lifetime determined by the residual gas scattering is longer than 1000 sec and it is expected that the average pressure should be lower than  $1 \times 10^{-5}$  Pa. Non-evaporable getter (NEG) pumps are mainly used with auxiliary ion pumps.

The construction of the DR was completed by the end of January 2017. It took more than two weeks from the start of the evacuation process from atmospheric pressure to the end of the activation of NEG pumps because of the small conductance of the beam pipes. The commissioning of the DR commenced in February 2017. When the beam was accumulated for the first time, the pressure exceeded  $1 \times 10^{-5}$  Pa with a beam current of ~1.5 mA. However, the pressure decreased quickly below  $1 \times 10^{-5}$  Pa. Subsequently, the pressure decreased gradually while the beam current was increased. The maximum stored current reached 11 mA on February 23. The residual gas composition of the pressure with the stored beam was typical of that expected for a vacuum system pumped by NEG pumps. Vacuum scrubbing has progressed smoothly thus far, and a beam lifetime of longer than 1000 sec was obtained with a stored beam current of 8 mA, when the beam dose was 0.7 Ah. The pressure distribution in the beam pipes was calculated using the Molflow+, and it is estimated that the photon stimulated desorption rate for CO at that time was reduced to  $\sim 1 \times 10^{-4}$  molec./photon. After beam tuning of the DR, the beam injection into the SuperKEKB commenced on March 27. No problems have been identified for the vacuum system to date.

**VT-TuP-7 Development of a Measurement System for Pressures in Vacuum Regions using an Optical Method, Yoshinori Takei, K Arai, H Yoshida, Y Bitou, S Telada, T Kobata, National Institute of Advanced Industrial Science and Technology (AIST), Japan**

Future primary standards of pressure based on optical measurement are being developed in national metrology institutes (NMIs) [1]. Pressure is proportional to refractive index according to both the equation of state of gas and the Lorentz-Lorenz equation. Refractive index can be measured using an optical method based on frequency measurement. Therefore, pressure measurements with a small uncertainty and a wide-range can be achieved. The purpose of this study is developing a pressure measurement system using an optical method for pressure range from 1 Pa to 100 kPa.

A Fabry-Perot interferometer was used for measuring the refractive index of the fulfilled gas in a chamber. The chamber, whose volume was around

7.6 L, was evacuated by a turbo molecular pump and a diaphragm pump. The background pressure and the pressure increase rate after closing the valve were respectively  $1 \times 10^{-3}$  Pa and  $2 \times 10^{-8}$  Pa/s, which cause one of uncertainty factors in a low pressure measurement. Pure nitrogen gas was used to fulfill the chamber. The pressure in the chamber was stabilized using a pressure balance from 10 kPa to 100 kPa or by simple accumulating from 1 Pa to 10 kPa. A commercially available external-cavity laser diode (ECLD) was used as frequency-tunable laser light source in order to measure wide-range frequency. The laser frequency was locked to a resonant line of an optical resonator installed in the chamber using the Pound-Drever-Hall (PDH) method. The locked frequency was measured by a beat signal comparing with the reference laser. Aluminum was used as the chamber material to reduce the temperature distribution around the optical resonator. Temperatures of the chamber were measured using several calibrated platinum resistance thermometers and several thermistors. The developed system measured fluctuations in the atmospheric pressure. The evaluated linearity and its uncertainty between refractive index of nitrogen and pressure from 1 Pa to 100 kPa gas are presented.

[1] Jay H. Hendricks et al., *X X I IMEKO*, 1574-1577, (2015).

**VT-TuP-8 Study on a Performance of a Sniffer Leak Detector based on EN 14624, Kenta Arai, H Yoshida, National Institute of Advanced Industrial Science and Technology (AIST), Japan**

Recent years, a portable type sniffer leak detector plays an important role for searching leaks of refrigerant gases from air-coordinators to prevent the global warming. In general, a standard leak, from which a small continuous gas (order of sub-g/year to ten-g/year) flows out, is used to check the performance of the leak detector for a reliable leak test. For the purpose, there are two methods to check the detector. One is that a probe of the detector is moved in front of the standard leak (method A) and the other is that the probe and an exhaust of the standard leak are closed and almost touched (method B). For method A, several standards have been published, i.e., SAE J1627:2011 and its related standards, EN 14624:2012[1], and ANSI/ASHARHE 173-2013. In those standards, the probe velocity and the distance between the probe and the exhaust of the standard leak are given and for EN 14624, 20 mm/s and 3 mm, respectively. For method B, it is explained in a manual of several leak detectors. In this study, we have measured the response of the leak detector against the standard leak by those two methods and compared their results.

A sniffer leak detector in which a pump equipped is tested. For method A, the detector was set on an electric actuator and its probe was set to face an exhaust of a standard leak. The moving velocity of the electric actuator was changed from 5 mm/s to 40 mm/s and the distance between the probe and the standard leak changed from 1 mm to 20 mm. For method B, the velocity was set at 0 mm/s and the distance at 0 mm, respectively. Prior to the tests, the leak rate of the standard leak was calibrated to be 5 g/year by a pressure rise method [2].

Both at the beginning and at the end of the experiments, the response of the leak detector was obtained by method B. Between them, the response of the leak detector was obtained by method A. The detector output obtained by method B between two measurements was stabilized within 10 %. For method A, the detector output was decreased with an increase of both the probe velocity and the distance from the probe to the standard leak. Above both the probe velocity of 20 mm/s and the distance of 10 mm, the detector output was disappeared. The detector output obtained by method B was about 5 times as high as that obtained by method A with the velocity of 20 mm/s and the distance of 3 mm given in EN 14624. Thus, for the properly use of the sniffer leak detector, it is important to clear the conditions for its performance check.

[1] EN 14624:2012 Performance of portable leak detectors and of room monitors for halogenated refrigerants.

[2] Kenta ARAI et al., *Metrologia* **51** (2014) 522.

**VT-TuP-9 Elimination of Electron-Beam-Induced Carbonaceous Contamination in SEMs and the new RGM 10100 NIST Contamination Testing Artifact, Andras Vladoar, K Purushotham, National Institute of Standards and Technology (NIST)**

Electron and ion beam-induced contamination could be a severe problem of scanning electron microscopes. The carbonaceous material deposited in a dynamic process of adsorption and desorption at the irradiated area can easily disturb imaging and lead to erroneous measurement results. The sources of contamination are usually both the SEM and the sample. Cleaning of the SEM can be carried out with a low-energy (oxygen) plasma

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cleaning process using commercial devices [1], but without a for-sure-clean sample, it is not possible to determine whether potentially time-consuming cleaning of the SEM is indeed necessary. The RGM 10100 sample with its associated cleaning and evaluation procedures, combined with appropriate cleaning processes offer an effective solution for this problem.

Figure 1 in the supplement shows three levels of SEM cleanliness. The energetic primary electrons can “purge” the center of the sample from carbonaceous contaminant molecules. The center brightening is due to slight oxidation caused by electron irradiation at 40 times the typical dose. With a clean RGM 10100 in a SEM there is no perceivable carbonaceous contamination even after many hours of continuous electron bombardment.

RGM 10100 can be cleaned with acidic piranha solution and can stay clean for months in a semiconductor grade plastic container. The necessary SEM cleaning time, depending on the cleanliness of the SEM, varies from 10 minutes to a couple of days. It is common that the contamination “comes back” after some time. As the SEM gradually becomes free of the source molecules of contamination, the time between needed cleanings could increase to months. RGM 10100 is available from the NIST Office of Reference Materials.

[1] <http://evactron.com> [<http://evactron.com/>], <http://ibssgroup.com> [<http://ibssgroup.com/>], <http://www.piescientific.com> [<http://www.piescientific.com/>]

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## **VT-TuP-10 PAL-XFEL Vacuum System, Donghyun Na, Pohang Accelerator Laboratory, Republic of Korea**

The Pohang Accelerator Laboratory X-ray Free electron Laser (PAL-XFEL) project was launched in 2011. This project aims at the generation of X-ray FEL radiation in a range of 0.1 to 10 nm for photon users with a bunch repetition rate of 60 Hz. The vacuum system of the machine consists of an injector section including an S-band photocathode RF gun, 10-GeV electron linac section based on S-band normal conducting accelerating structures and a 150-m long out-vacuum undulator system. Narrow and long aluminum chamber fabricated special extrusion method and inner surface of undulator vacuum chamber treated mirror-like polishing for low ac electrical resistance and wake-field effect. This paper presents the design of the PAL-XFEL vacuum system and achieved results.

## **VT-TuP-11 Extreme 2 Million Liter/sec Hydrogen Pump Speed Measurements of C-2W Divertors, Ernesto Barraza-Valdez, A Van Drie, TAE Technologies**

TAE Technologies (TAE) has completed building and commissioning its latest machine, C-2W. C-2W has four 15m<sup>3</sup> divertors which require the capability to pump out H<sub>2</sub> neutral particles at a rate of 2,000 m<sup>3</sup>/s and D<sub>2</sub> neutral particles at 1,500 m<sup>3</sup>/s. To achieve this, TAE has developed LN<sub>2</sub> cooled titanium getter cryobox pumps each with a pump speed of approximately 110 m<sup>3</sup>/s and 85 m<sup>3</sup>/s for hydrogen and deuterium respectively. These cryoboxes are arrayed in the divertors to achieve the high pump speeds required to keep the plasma conditions optimal during C-2W operations. Experimental pump speed and density measurements using fast ion gauges and residual gas analysis of C2W's four divertors will be presented.

## **VT-TuP-12 KICT Dirty Thermal Vacuum Chamber: design, fabrication, and performance test, T Chung, Korea Institute of Civil Engineering and Building Technology, Republic of Korea; Jong Yeon Lim, Korea Research Institute of Standards and Science, Republic of Korea; Y Yoo, H Shin, Korea Institute of Civil Engineering and Building Technology, Republic of Korea**

KICT is designing and building a large thermal vacuum chamber with a simulant regolith test bed inside, named Dirty Thermal Vacuum Chamber (DTVC), capable of performing characteristics evaluation of extraterrestrial exploration drill tools, rovers, and 3D printing tools, as well as investigating the physics and effects of thermal radiation. The DTVC system consists of a D-shape main chamber,  $\phi$  4 m x 4 m, and a preliminary chamber of  $\phi$  4 m x 2 m enabling of preparing and conditioning samples. The extension of the simulant bed to 6 m in length is also possible with opening the gate valve between two chambers for a rover maneuvering test through both chambers.

To successfully establish the DTVC system, a vacuum, cryogenic, and anhydrous (no water) soil environment, simulation of the lunar and Mars surfaces was first proposed with physics requirements of -190 °C ~ 150 °C in the pressure range of 10<sup>-4</sup> mbar ~ 10<sup>-5</sup> mbar. In order to realize the desired pressure of 5 x 10<sup>-8</sup> mbar without any components inside, the baseline exhaust system has been equipped with two 2,000 L/s TMPs and

two 28,000 L/s cryopumps as well as one 3,000 m<sup>3</sup>/h roots pump for roughing and two 300 m<sup>3</sup>/h dry pumps for backing. The samples then can be exposed to thermal radiation on the simulant regolith with UHV background vacuum quality.

Since about 25 tons of simulant regolith are necessary for the bed, the regolith outgassing rate of <5 x 10<sup>-8</sup> mbar.L/(s.g) is to be strictly regulated to satisfy the physics requirement. For this purpose a smaller scale chamber, called Pilot DTVC, was simultaneously built to realize the outgassing rate of the regolith. An amount of 125 kg of KLS-1, simulant regolith developed by KICT, was pumped down to about 0.1 mbar to remove water content down to <1 x 10<sup>-4</sup> mbar.L/(s.g). Previous TDS study of the KLS-1 regolith shows the most content of the regolith ingredient adsorbed is water, and the H<sub>2</sub>O dose was about 1.2 x 10<sup>5</sup> mbar.hours during nine month exposure to 50 % RH ambient environment at 300 K average.

This presentation will focus on the brief introduction of the vacuum architecture, and design implementations of the DTVC and Pilot DTVC system for both qualitative and quantitative analyses to satisfy all the strict vacuum requirements.

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