Vacuum Technology Division
Room 7 & 8 - Session VT-WeM

Transfer and Ultraclean Systems, Particle Control, and History

Moderators: Jason Alfrey, Vacuum Technology, Inc., Marcy Stutzman, Jefferson Lab

8:00am **VT-WeM-1 Applications and Challenges of UHV- and Cryo Transfer of Samples Between Independent Analytical Systems**, ***Urs Maier***, *S Köster, D von Gunten,* Ferrovac GmbH, Switzerland*; S Yoshizawa, T Uchihashi,* National Institute for Materials Science, Japan*; S Rauschenbach,* Max-Planck-Institute for Solid State Research, Germany **INVITED**

To answer demanding questions in the characterization and analysis of sample surfaces a variety of high performance atomically precise methods are available. Molecular beam epitaxy (MBE), focused ion beams (FIB), electrospray ion beam deposition (ES-IBD) or even vitrification of biological samples can fabricate surfaces of great complexity with atomic/molecular precision. Similarly, analytical methods like scanning probe microscopy (SPM), electron microscopy (SEM/TEM), photo- or electron spectroscopy (e.g. XPS), low energy electron holography or secondary ion mass spectrometry (SIMS) provide spatial or chemical insight of highest precision. A combination of these methods is often desirable while these methods require a clean environment, typically ultrahigh vacuum (UHV, 10^{-10} mbar) and sometimes cryogenic conditions to maintain the sample in a state that allows for meaningful results.

A sample transfer with undisrupted vacuum and cryogenic conditions cannot always be achieved via a direct linkage between the instruments. More complex combinations, involving many different methods, are often impractical. The ability to transfer samples under well controlled environmental (UHV) and thermal conditions between independent analytical systems therefore greatly extends possible applications of the instrumentation.

Here we present the NexGeneration UHV suitcase system that allows to transport samples in UHV environment between instruments of even far separated facilities. The system consists of a lightweight and readily transportable vacuum chamber (the suitcase) which is actively pumped by a combination of a getter/ion pump (SAES, NEXTorr) using a battery powered controller which allow for up to 2 days off-grid operation. The transfer is facilitated via loadlocks attachable to arbitrary systems. The suitcase is of modular configuration allowing the addition of sample storage positions, adaptation to different carriers, as well as choosing from different types of sample transporters to handle the sample. Above all, a cryogenic variant of the suitcase has recently been developed, in which samples are actively cooled down to -190°C on a liquid nitrogen cooled stage situated within a thermal shield.

In the presentation we show our implementation of a sample transfer with the NexGeneration UHV suitcase system and show configurations established for different research groups. We further present data measured from samples transported within our suitcase that shows the cleanliness expected from samples that were maintained in UHV, clearly showing the capability of our system for a large variety of applications.

8:40am **VT-WeM-3 Ultra-clean Sample Transportation in an EUV Exposure System**, ***Freek Molkenboer***, *N Koster, A Deutz, B Nijland, P Kerkhof, P Muilwijk, B Oostdijck, J Westerhout, C Hollemans, W Mulckhuyse, M van Putten, P van der Wall, A Hoogstrate, H Diesveld, A Abutan,* TNO, Netherlands

In 2015 TNO started the design of a new Extreme Ultra-Violet (EUV) exposure facility, called EUV Beam line 2 (EBL2). EBL2 will be a publicly accessible test facility for EUV lithography related research and qualification. The realisation of the EBL2 started at the end of Q1 2016. On December 7th 2016 the important milestone " First light" was accomplished.

EBL2 is designed to be able to load a wide range of sample types, including the EUV industry standard 6" reticles. To achieve this, all the samples are loaded using the SEMI standardised EUV dual pods.

Sample loading for the EBL2 facility starts at the Atmospheric Handler. The EUV dual pod is opened, and the Atmospheric Handler robot transfers the sample to the load lock of the EBL2 system. The Atmospheric Handler has several ultra-clean environments to limit the particle contamination on the samples.

After the load lock is evacuated to vacuum, the robot of the Vacuum Handler will transport the sample to the operator-selected module of the EBL2 system. Besides the load lock, the Vacuum Handler connects to the Expose Chamber, an XPS, and two chambers that are used for storage and cleaning of samples. In the Exposure Chamber samples can be exposed to EUV irradiation in various controllable gas environments. An XPS is available for surfaces analysis after an experiment while maintaining vacuum. The handling between the chambers is fully automated with multiple checks to ensure sample safety.

When a sample is transported to the Exposure Chamber the sample must be flipped from a horizontal loading position to a vertical mounting position. This is due to the design constraints of the EUV source and illumination module of the EBL2 system. After this flip from horizontal to vertical the sample must be positioned and clamped against the cooled Sample Chuck.

Both the flip and the clamping motions use pneumatic actuated bellows that are located inside the Exposure Chamber.

The Sample Chuck positions the sample in the EUV irradiation spot across the entire 6" reticle. The movement of the Sample Chuck is accomplished with a Hexapod that is located outside the vacuum. The vacuum barrier between the hexapod and the Sample Chuck is a large edge welded bellow.

During this presentation we will discuss our implemented design solutions for sample handling in ultra-clean vacuum. The objective of the design and implementation are to maintain the stringent vacuum and particle requirements for these kind of experiments.

9:00am **VT-WeM-4 Oxidation and Contamination Monitoring Methods for Air Sensitive Materials Transfer: From Glove Box to UHV Surface Analysis**, ***Hugo Celio***, *K Ohlinger,* University of Texas at Austin

The performance of lithium ion-batteries is steadily improving but there is still need for higher energy density and cycle life in consumer applications. An ex situ investigation of the composition is crucial for investigating performance issues. After a cycling period, the cathode (or anode) material is extracted from a battery coin cell under an argon environment of a glove box. However, a glove box also contains traces of O_2 and H_2O in the 1-part-per-million range, an unknown amount of adventitious hydrocarbons and inorganic impurities. These traces of oxidants and contaminants are carried along as battery materials are transferred from a glove box to a UHV chamber for surface analysis.

An interface designed to transfer air sensitive materials from a glove box to an ultra-high vacuum (UHV) chamber for surface analysis was previously presented[1]. This interface is called an interface for pressure-to-vacuum environmental sample transfer, or IP-VEST. It is coupled to a UHV chamber equipped with X-ray photoelectron spectroscopy (XPS). The IP-VEST has a built-in method for transfer reliability. However, there is no known method to monitor the degree of oxidation to air sensitive materials from exposure to trace levels of oxidants in argon.

We selected silicon, silver, tin and lithium as a set of reference materials to monitor their surface oxidation and contamination from exposure to trace levels of oxidants, organic, and inorganic contaminants during the environmental transfer of air sensitive battery materials as described above. The preparation procedure for the reference materials is a simple mechanical step which is carried out in the glove box. We focused on evaluating the oxidation rates of clean Si, Ag, Sn and Li with respect to travel time, which is dictated by distance between the glove box and the IP-VEST/UHV chamber. At UT, five glove boxes are located within short distances of each other, entailing a preparation and travel time of less than 30 minutes. Upon arrival to the UHV chamber, the capsule, containing the air sensitive battery materials and reference materials, is coupled to the load-lock of the IP-VEST. This transfer process requires 1 hr. for samples from atmospheric argon pressure to high vacuum conditions. Based on XPS data, the surface oxidation of the reference materials significantly varies but yield a range of oxidation rates. These oxidation rates can be compared to the oxidation rates of some battery materials. The environment of the glove boxes is effectively inert for most cathode materials but insufficient for highly reactive battery materials like lithium.

[1] AVS 2015 and U.S. Patent Application Serial No. 14/445,650

9:20am **VT-WeM-5 Particle Contamination Control in the Accelerator Vacuum Systems of the European XFEL**, ***Lutz Lilje***, *S Lederer,* DESY, Germany **INVITED**

For the European XFEL accelerator vacuum about 1,5 km of vacuum system have been assembled with procedures that result in a low particulate contamination inside of the vacuum components. For this mechanical

design aspects as well as cleaning and installation procedures needed to be adapted to a large variety of beam line components. An example is the development of dedicated clean rooms for the installation of components in the accelerator tunnel.

The experience with the installation is presented as well as preliminary results from the commisioning of the accelerator. An initial correlation of difficulties observed during the installation process with performance of modules will be discussed.

11:00am **VT-WeM-10 Development, Solution of Design Issues, Final Design and Performance of an Electrostatic Triode Getter-Ion Pump, 1967-1973***, Paul Arnold,* MKS Instruments, Inc. **INVITED**

History of a completed commercial electrostatic triode getter-ion pump, where ion pumping and getter pumping were separated allowing preservation of the getter at UHV, will be presented. Also covered will be the solutions of combining high temperature, high voltage, high getter sublimation rate, and ultra-high vacuum in one pump design from the late 1960s. The getter was operated with active gas pumping speed as a direct function of the power to the getter and was independent of the ion pumping, allowing preservation of the getter material at UHV while maintaining full ion pumping speed. The physical electronics of a hot filament ion pump design with four pumping cells, each with dual filaments, will be shown. The successful joining and assembly of many refractory materials, some at temperatures reaching 1600 Celsius, will be described. The pump operates with electrodes at voltages up to 4000 volts in the environment of a titanium sublimation rate up to 0.02 grams per hour while maintaining adequate resistance values of the many insulators. Pumping speeds for various gasses will be displayed, along with pumpdown curves for many gasses with a gas type comparison to a sputter-ion pump from the same 1960s era. Automatic turn-on and turn-off pressure indications were provided by a heat-loss gauge with full-scale resolution of 10 millitorr. This Invited Talk is part of the AVS History Committee's endeavor to preserve and promote our vacuum technology history.

11:40am **VT-WeM-12 The Modern View of the Vacuum***, H. Frederick Dylla,* American Institute of Physics

The concept of the vacuum has evolved from ancient to modern times. Ancient Greeks did not believe in the concept of vacuum-empty space in which nothing exists. With their early formulation of atom-like particles, they believed that matter was completely space filling. As civilization moved into the "Enlightenment" and the early industrial age, a practical definition of vacuum became any space evacuated to a pressure less than ambient. This is still a practical definition of vacuum in contemporary times, where state-of-the-art techniques can produce extreme vacuum levels-approaching matter densities of less than a molecule/cm^3. However, from the standpoint of contemporary physics, we have moved back to a view that the vacuum is not empty space devoid of content. Paul Dirac's theory of Quantum Electrodynamics, the most precisely experimentally benchmarked theory in science, portrays empty space as being filled with quantum fluctuations: virtual particle-antiparticle pairs appearing and disappearing on extremely short (Planck) time scales. The present status of cosmology research adds additional complexity to the concept of a perfect vacuum. Quantum fluctuations underpin Alan Guth's inflationary model of the universe's expansion following the primordial Big Bang. His widely accepted analysis explains the high uniformity of matter density in the observable universe-a part in 10^4. Quantum fluctuations in space drove an immense (10^{28}) expansion of the primordial universe using the latent energy in a so-called false vacuum. Over the last two decades additional observations of the universe's expansion rate, have shown that the visible components of the universe (matter and radiation) account for only about 1% of the content- 30% resides in dark matter and 70% in dark energy. Characterizing these latter two components remains on the forefront of modern physics research, and clearly a perfect vacuum is far from empty.

12:00pm **VT-WeM-13 History of Very Thick Film and Bulk Sample Group IIIB, IVB, VB and Rare Earth Materials for Various Vacuum Applications***, James L. Provo,* J.l. Provo Consulting

History of Very Thick Film and Bulk Sample Group IIIB, IVB and Rare Earth

Materials for Various Vacuum Applications

James L. Provo [a]

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Thick occluder films of hydride materials are extremely hard to produce without

flaking or cracking. This paper discusses methods of how to prepare thick films

and bulk samples (i.e., rods and wires) for many applications including accelerator

research for cancer therapy, intense neutron source, and particle-beam fusion

diagnostic beam focusing studies. These thick films ~ (≥ 5,000 to 15,000 nm

thick) of various hydrides are sensitive to oxidation and are easily contaminated by

improper handling. They must be specially prepared to reduce internal stresses due

to temperature variations during processing, and stresses due to hydriding and to

substrate configuration (i.e., curved surfaces). This paper will discuss techniques

developed at the General Electric Neutron Devices Department (GEND), in Largo, FL,

in the mid-1970's to the late 1990's to produce stress free and thus flaking and crack

free samples of thick films and bulk samples. Items studied include, Er, Sc, and Ti

thick film hydrides on a Cr underlay, on various substrates, bulk rod samples (0.635

cm O.D. by 2.54 cm long) for basic material heat capacity and thermal diffusivity

studies as a function of hydride loading, Nb and V wires 10 and 20 mil O.D. by 5.08

cm long in bundles of ~30 wires for neutron vibration spectra studies and 20 mil O.D.

by 1.27 cm long Ti wires for mass spectrometer calibration studies. Film samples were

prepared by standard E-beam evaporation techniques and then non air-exposure loaded.

Bulk samples were cleaned, weighed, and then loaded with a Sievert's precise gas

quantity loading system. Special processing to accomplish flake and crack free samples,

included heating sample substrates for thick films to 450°C, evaporating at a controlled

rate of 10 nm/min.,which takes ~ 8.3 hrs. for 5,000 nm films and ~25 hrs. for 15,000

nm films, followed by non-exposure loading (i.e., leaking D_2 or T_2 gas into the loader) at a

rate of 1Torr/hr. until 50 Torr is reached, which will take ~2-days, holding at temperature

and pressure for ~ 8hrs, then cooling down at a rate of 1°C/ min.from 450°C to room

temperature. Er films are cooled to ~ 320°C, then gas is removed to the source bed to

prevent trihydride formation, before cooling to ~ (25°), which will take ~ 7.5hrs. Using

the process described, very successful results were obtained.

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