Vacuum Technology
Room 201 - Session VT-WeM

Sorption Processes and Leak Detection
Moderator: J.F. O'Hanlon, University of Arizona

8:20am **VT-WeM1 The United States Constitution and Vacuum Technology**, ***C.R. Tilford,*** Consultant, U.S. **INVITED**

The Charters of Freedom, the official copies of the United States Declaration of Independence, Constitution and Bill of Rights, were hermetically sealed in 1952 in glass and lead encasements filled with humidified helium. Deterioration of the inside glass surfaces has become evident in recent years, and is believed due to elevated water vapor concentrations, which can also be a direct threat to the parchment documents. The National Archives and Records Administration and the National Institute of Standards and Technology have undertaken to construct new encasements that will keep oxygen concentrations below 0.5% over 100 years, maintain a stable humidity level, and include mechanical and broadband optical access for gas sampling and spectroscopic monitoring. Prior to disassembly of the existing encasements, gas samples are extracted for humidity measurement and mass spectrometric analysis. This project presents several difficult vacuum technology challenges, including hard-sealing large glass windows to lightweight structures (for public viewing of the documents), helium leak testing the sealed encasements without evacuation, measuring humidity of small-volume gas samples, and extracting clean samples from the small-volume solder-sealed encasements. In all cases the solutions are constrainted by the absolute necessity to avoid perturbing the documents or over-stressing the lightweight encasements. To date, the gas sampling and reencasement process has been carried out for three pages of the Constitution. This talk will describe the vacuum techniques used and present the experiences and results to date.

9:00am **VT-WeM3 Measurements of Photon Stimulated Desorption from Thick and Thin Oxide of KEKB Collider Copper Beam Chambers and a Stainless Steel Beam Chamber@footnote 1@**, ***C.L. Foerster,*** *C. Lanni,* Brookhaven National Laboratory*; K. Kanazawa,* KEK, Japan

Photon Stimulated Desorption(PSD) from KEKB factory copper chambers was measured at the National Synchrotron Light Source(NSLS), located at the Brookhaven Laboratory. The KEKB is an asymmetrical collider recently constructed in Ibaraki, Japan. The collider utilizes two UHV ring chambers, one for a 3.5 GeV positron beam and the other for a 8 GeV electron beam, for B-meson studies. Two each, one meter long, 94mm inner diameter, chemically deoxidized copper beam chambers with conflat end flanges, were provided by KEKB to the NSLS, for measurment of PSD. PSD and specular reflection were measured on NSLS VUV ring beamline U9a. It has been reported that a high temperature air bake on large stainless steel vacuum systems reduces the need for a vacuum bake. To determine the effect of surface oxide, following the initial PSD measurment, the chambers were chemically cleaned and then oxidized in air for a week at 250 °C. PSD was remeasured after the air bake. A similar process and measurment was performed on a stainless steel beam chamber, with the exception of oxidation at 450°C. After the chambers were installed on beamline U9a, they were exposed to a minimum of 10@super23@ photons direct from the source having a critical energy of 595 eV, striking at an incident angle of 100 mrad. The major PSD yields for hydrogen, carbon nonoxide, carbon dioxide, methane, and water vapor are reported as a funtion of accumulated photon flux, incident surface oxide, and chamber preparation. The results are compared with other PSD measurments on NSLS beamlines and those of other laboratory publications for copper and stainless steel. @FootnoteText@ @footnote 1@Work performed under the auspices of the U.S. Department of Energy, under contract DE-AC02-98CH10886.

9:20am **VT-WeM4 Study the Exposure Dose-Dependent Photon Stimulated Desorption Phenomena**, ***G.Y. Hsiung,*** SRRC, Taiwan*; K.-Y. Young,* Institute of Nuclear Science, NTHU, Taiwan*; Y.J. Hsu,* SRRC, Taiwan*; J.-R. Chen,* SRRC and Institute of Nuclear Science, NTHU, Taiwan

The photon stimulated desorption (PSD) is studied by using the synchrotron radiation white light of critical energy at 2.14 keV from the 1.5 GeV Taiwan Light Source (TLS). The samples of various materials, e.g. aluminum alloys, OFHC, etc. are installed in a chamber at a beam line of SRRC for exposure measurement. The sample is water cooled during the exposure to the synchrotron light that the variation of temperature on the surface is < 0.2 degree C. A quadrupole mass spectrometer is used to measure the desorbed gas molecules by PSD in the exposure chamber. The result shows a decrease of the PSD-rate for each gas after a long time exposure of photon beam on the samples. The measured photoemission yield on the sample during exposure also decreases. The curves of pressure rise of each PSD-gas versus the time during the exposure show various kinds of trend with different time lag of the peak. The desorbed molecules including H2O, O2, CO, and CO2, have the time lag longer than those of H2 and hydrocarbon molecules. The longer beam dose exposed on the sample, the longer time lag of PSD peak trended. The phenomenon of exposure dose-dependent PSD relates to the surface condition of the exposed samples. A Secondary Ion Mass Spectroscopy (SIMS) system is built in the exposure chamber to assist the in-situ surface characterization. The correlation between the yield of both PSD and photoemission and the surface concentration during and after the exposure will be compared. The phenomenon of exposure dose-dependent PSD is described and compared.

9:40am **VT-WeM5 Study of Desorption of Carbonaceous Gas Molecules from Copper Surfaces under Electron Bombardment**, ***M. Nishiwaki,*** The Graduate University for Advanced Studies, Japan*; S. Kato,* KEK, Japan

Adoption of oxygen free copper has been recently begun to vacuum chambers of particle accelerators such as the KEKB accelerator that is a two ring electron-positron collider. Due to irradiation of the energetic particles and/or exposure to residual gas, however, the copper surface composition should be easily altered and consequently would give instable vacuum property because the passive surface cannot form on copper material which is different from other materials with passive surface such as stainless steel and aluminum. In this study, we aim to make clear about the mechanism about the adsorption and desorption on the copper surface using some isotope gas species. The copper surfaces to which some surface treatments were carried out were exposed to isotope gas species of @super 13@C with a known quantity in an ultra high vacuum. Afterwards, electron stimulated gas desorption (ESD) rates from these sample surfaces were measured using throughput method with a calibrated residual gas analyzer quantitatively. By using the isotope gas, desorbed gas under electron bombardment from the surface on which @super 13@C related gas molecules adsorb can be distinguished from desorbed @super 12@C related gas molecules which consist of diffused carbon atoms from the bulk since @super 13@C atoms do not exist in the bulk. Preliminary experimental results showed that enhancement of carbon diffusion to the surface under the electron bombardment occurred at a high sample temperature. This might suggest that the origin of carbon in the carbonaceous desorbed gas molecules such as CO and CH4 is not the top surface but the bulk possibly in a electron penetoration depth. Surface characterization of the copper materials will be also done using x-ray photoelectron spectroscopy in the adsorption and desorption processes of the isotope gas.

10:20am **VT-WeM7 Vacuum Characteristics of Sprayed Metal Films**, ***M. Minato***, *H. Iwamoto,* Vacuum Metallurgical Co., Ltd., Japan

The need to reduce particles in semiconductor equipment or data storage equipment is increasing. In recent years, barrier and glue layers on shielding have been used to reduce particles generated in physical vapor deposition equipment. For this purpose, spray coatings are applied to shield components after contaminations are removed. Although the sprayed metal films are exposed to vacuum in a vacuum chamber, their vacuum characteristics have seldom been investigated. The conventional spray coatings are usually done under atmospheric conditions. In order to obtain a layer that has a better vacuum characteristic, spray coating in a chamber filled with an inert gas was developed. Outgassing characteristics of sprayed aluminum films and titanium films were investigated by using the conductance modulation method. Sprayed titanium film produced in an inert gas system showed a lower outgassing rate than that produced in an atmospheric condition.

10:40am **VT-WeM8 Edison's Vacuum Coating Patents**, ***R.K. Waits,*** Consultant

Among the more than 1,000 patents bearing Edison's name are several for vacuum coating processes. In 1894 Edison was granted a patent on the "Art of Plating One Material on Another." The patent described coating by evaporation in a vacuum by direct resistance heating or arc heating, using a continuous current. Edison called the process "electro vacuous deposition." He prophetically wrote that "the uses of the invention are almost infinite". Edison also employed sputter deposition. In 1900, Edison applied for a patent on a "Process of Coating Phonograph Records." Issued in 1902, the patent describes using a "silent or brush electrical discharge" produced by an induction coil. Edison had found a way, perhaps accidentally, to use

high-voltage alternating current to deposit a metal; the pressure was higher and deposition was by sputtering rather than evaporation. The National Phonograph Company, one of Edison's many enterprises, used the sputtering process to deposit a thin layer of gold on wax phonograph cylinder masters that could then be electroplated to form molds to mass-produce celluloid duplicates. The resulting cylinders were touted as "Gold Moulded." The method was used for 20 years, from 1901 to 1921. It enabled the reproduction of cylinder grooves less than 0.001-inch deep at a density of 200 grooves per inch. From 1913 to 1921, 10-inch-diameter Edison Diamond Disc phonograph records were made using the same method. Sputtering was abandoned as it could not be scaled up to produce the 12-inch discs that were introduced in 1927.

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