Wednesday Morning, November 6, 2002

Vacuum Technology
Room: C-104 - Session VT-WeM

Outgassing
Moderator: L.A. Westerberg, The Svedberg Laboratory

8:20am **VT-WeM1 Influence of Thermal and Surface Treatments on the Outgassing Of Austenitic Stainless Steels Studied by Thermal and Electron Stimulated Desorption**, *C. Benvenuti, **P. Chiggiato**, G. Chuste, I. Wevers*, CERN, Switzerland **INVITED**

Austenitic stainless steel is at present the most widely used material for UHV applications. In order to improve its vacuum behavior, several post-production treatments are usually applied. Surface chemical and electrochemical processing are used to reduce surface contamination and/or roughness, while heating, either under vacuum or in air, is applied to decrease hydrogen thermal outgassing. The effect of these treatments on the content and on the binding state of surface and bulk gases has been studied by thermal desorption spectroscopy and electron stimulated desorption. The enrichment of hydrogen induced by surface electro-polishing and the effect of the residual hydrogen pressure during vacuum firing have been studied in detail.

9:00am **VT-WeM3 Comparison of Outgassing Rates of Bare and TiN-coated Stainless Steel Vacuum Chambers***, *P. He, H.C. Hseuh, M. Mapes, **R. Todd**, D. Weiss, D. Wilson*, Brookhaven National Laboratory

The stainless steel vacuum chambers of the 248m accumulator ring of Spallation Neutron Source (SNS) are to be coated with ~ 100 nm of TiN to reduce the secondary electron yield. Each SNS chamber is approximately 4m long and over 20cm in diameter. The coating is produced by DC magnetron sputtering using a long cathode imbedded with permanent magnets. Reports in literature suggest that the potential benefit of a TiN coating as a hydrogen permeation barrier that reduces the ultimate outgassing rate. The outgassing rates of several SNS chambers were measured with and without TiN coating of various thicknesses, and before and after in-situ bake. No improvements in outgassing were observed with the coated chambers. The results of the coated chambers were compared with the surface structure and thickness of the TiN coatings as analyzed using AES and SEM. A correlation between film density and outgassing rate was found. *Work performed under the auspices of the U.S. Department of Energy.

9:20am **VT-WeM4 Degassing of Static Expansion Vacuum Gauge Calibration Chamber**, ***J. Setina***, Institute of Metals and Technology, Slovenia

Laboratory of Pressure Metrology at the Institute of Metals and Technology is developing a static expansion system for calibrations of vacuum gauges. The gasses evolved from the chamber walls significantly affect the accuracy of generated calibration pressure at low pressures below 1mPa. The chambers of the expansion system were designed in accordance with established ultrahigh vacuum practice and are made from stainless steel. We studied the outgassing characteristics of the large calibration chamber by the gas accumulation method. The chamber was sealed-off from the pump and the rate of pressure rise was measured using the spinning rotor gauge (SRG). From this the total outgassing rate can be determined. At the end of the accumulation period the chamber was evacuated again. During evacuation the composition of the accumulated gas was dynamically analyzed with the quadrupole mass spectrometer. The method of dynamic gas analysis will be described and outgassing of unbaked and baked chamber will be compared. Bake-out temperature was gradually increased from 60 °C to 250 °C. By bake-out at 100 °C for 50 hours we effectively removed water vapour and other adsorbed gases and the main outgassing component became H2 at a rate of 1x10-11 mbarls-1cm-2. Final outgassing rate after 160 hours of bake at 250 °C was 2.4x10-13 mbarls-1cm-2. Measured H2 outgassing rate after bake-out at 100 °C increased by a factor of 3 compared to the initial value of unbaked surface. This indicates the importance of the adsorbed layer on stainless steel surface on the kinetics of H2 evolution. The diffused hydrogen atoms from the bulk must recombine into H2 molecule in order to be de-sorbed. Other adsorbed gasses can hinder the recombination of hydrogen atoms.

9:40am **VT-WeM5 Organic Contaminants Adsorption Behavior on Silicon Wafer Surface Under Reduced Pressure**, ***T. Hayashi**, T. Kawaguchi, N. Tanahashi,* Tohoku University, Japan, *M. Saito, K. Suzuki,* Tokyo Electron Ltd., Japan, *Y. Wakayama,* Taisei Corporation, Japan, *Y. Shirai, T. Ohmi*, Tohoku University, Japan

It is known that organic contaminants on silicon wafer surface cause many detrimental effects, such as degradation of gate oxide integrity, yield losses, and so on. To prevent that, closed manufacturing system has been proposed. However, these are considered only for outside of process chamber under atmosphere pressure. And now, the low-pressure processes such as CVD, sputter, dry etching, etc. are routinely used under several hundred mTorr or less. The organic contamination behavior on silicon wafer surface in low-pressure chamber has not been reported yet. In this study, we clarified the organic contamination behavior on silicon wafer surface under reduced pressure. We have researched the following three results; 1) The amount of organic compounds adsorbed onto a silicon wafer stored in a chamber under reduced pressure was much more than that stored under atmosphere pressure. 2) The volatile level released from pure n-Eicosane, typical hydrocarbon contaminant, does not depend on the pressure. 3) The adsorption behavior for the amount of volatile from n-Eicosane adsorbed onto silicon wafer surface coexisted with n-Eicosane in the low-pressure chamber is in accordance with Langmuir model of monolayer adsorption and the amount of adsorption saturated is in inverse proportion to pressure. In conclusion, we clarified that the inner pressure of a chamber become lower, the partial pressure of released organic compounds from chamber components becomes higher, that is, the contact frequency between organic compounds and silicon wafer surface become higher. As a result of that, the much amount of organic compounds is adsorbed onto silicon wafer surface.

10:00am **VT-WeM6 Study on Desorption of Carbonaceous Gas Molecules from Copper Surfaces under Electron Bombardment and the Surface Characterization**, ***M. Nishiwaki***, KEK, Japan, *S. Kato*, KEK & The Graduate University for Advanced Studies, Japan

In particle accelerators, the carbonaceous gas desorption is generally observed as a result of the energetic particle stimulation at ducts and r.f. component surfaces during the operation and would deteriorate the beam performance. Therefore we aim to understand origin of the desorbed carbonaceous gas from the copper surface using carbon isotope. The passive layer cannot be formed at the surface of the oxygen free copper adopted to the beam duct. Since the surface should be easily altered due to irradiation of energetic particles and/or exposure to residual gas, the carbonaceous gas desorption mechanism from the surface is of high interest. In this study, we focused on the electron stimulated desorption (ESD) from the copper surface and its in-site surface characterization by x-ray photoelectron spectroscopy and Auger electron spectroscopy. For the surface cleaning and initializing, the copper samples were sputtered with Ar$^+$ ion beam and annealed. Exposure of ^{13}C isotope gas and ^{13}C ion implantation were done to the samples with the known quantities respectively. Afterwards, ESD rates from the samples were measured using throughput method with a calibrated residual gas analyzer quantitatively. By using the isotope, influences of ^{12}C in the residual gas and the bulk of copper can be eliminated during observation of desorbed gas from the sample under electron bombardment. The ESD of ^{13}C related gas species from the surface exposed to $^{13}CO_2$ were not observed. In the experiments of ^{13}C implanted samples, the ESD of ^{13}C related gas species with a low current density were not observed either at the room temperature. However the ESD rates of ^{13}C related gas species from the heated ^{13}C implanted sample around 65 degrees C were as high as the thermal desorption rates at around 250 degrees C. These results showed that the diffusion of implanted ^{13}C from the bulk was enhanced by heating and/or electron bombardment depending on the beam current density and might suggest that origin of the desorbed carbonaceous gas is not the top surface but the bulk possibly in a range of the electron penetration depth.

10:20am **VT-WeM7 Solubility and Diffusion of Hydrogen in Ordered Metals**, ***P. Repa**, L. Peksa, T. Gronych, R. Ulman*, Charles University, Czech Republic

In the materials with ordered structure, such as the nanocrystalline and gradient materials, a relatively great part of the material is influenced by the interfaces between the crystals or between the components. As the interfaces differ from the bulk by the density and the topology of atoms, physical properties of the materials are influenced. As a result, the metals of that structure exhibit changes not only in ductility and other mechanical properties but also in the electric and magnetic properties as well when exposed to hydrogen. That's way knowledge of hydrogenation kinetics of the materials have become in the great interest recently. Since the measurement of the solubility and diffusion coefficient of hydrogen in the ordered metals is complicated due to mechanical properties of the measured samples, requirements on the experimental set up and measuring procedure is discussed. Results of the measurements are presented in which a sample was exposed to the hydrogen at elevated temperature in a filling chamber for a satisfactorily long time period to be saturated. Then the saturated sample was gradually heated up in a UHV system and the variations of the total pressure and partial pressures of several selected gases were registered. The total amount of gas dissolved in the sample, the values of solubility and diffusion constants could be estimated by an analysis of the obtained experimental data. A strong dependence of the solubility of hydrogen on the structure and composition of structured metals was discovered.

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