Vacuum Technology Division
Room 610 - Session VT-ThM

Outgassing, Leaks, and Mass Flow Controllers

Moderator: W. Weed, Sandia National Laboratories

8:20am **VT-ThM1 A Comparison of Outgassing Rates from Stainless and Carbon Steels**, ***H.F. Dylla,*** Thomas Jefferson National Accelerator Facility; *W.R. Blanchard,* Princeton University

Various types of stainless steels (ANSI type 300 series) are the most commonly used materials for the construction of high and ultrahigh vacuum vessels and associated vacuum hardware. As a result of this widespread use, numerous outgassing studies of stainless steels have been published in the literature. The available information on outgassing from carbon steels is relatively meager. Carbon steels are usually not considered for use in high vacuum systems because of the propensity for corrosion (oxidation) in ambient environments and the assumption that standard surface preparation techniques would be less effective on carbon steels in comparison to 300 series steels. During consideration of a number of vacuum applications of carbon steels we reviewed the outgassing literature and found that an often quoted measurement significantly overestimates the outgassing from properly cleaned material. We describe outgassing measurements we have performed on both types of steel in order to get a direct comparison of the materials on the same apparatus and using the same surface preparations techniques. Results show that the short-term (<100 hr) outgassing rates from clean carbon steel are only a modest factor (times two to four) larger than from similarly prepared stainless steel. The increased outgassing rate appears to be in direct proportion to the passivation oxide layer thickness. @FootnoteText@ This work was supported by US DOE Contract No. DE-AC02-76-CH-03073.

8:40am **VT-ThM2 Experiments with Thin-Walled Stainless Steel Vacuum Chamber**, ***V. Nemanic,*** Institute of Surface Engineering and Optoelectronics, Slovenia; *J. Setina,* Institute of Metals and Technology, Slovenia

The application of reasonably thin stainless steel wall was suggested recently as alternative approach in construction of UHV chambers, since the hydrogen outgassing rate can be decreased to the level required to reach UHV and EXV much easier. Experimental stainless steel chamber (AISI type 304, volume 13 l) with uniform wall thickness (0.6 mm) has been constructed. It was equipped with a miniature ion-getter (IG) pump and spinning rotor gauge (SRG) thimble. The chamber was pumped down and initially degassed by moderate bake-out (2.5h, 150 deg.C) by a turbomolecular pump system. Gas accumulation method was applied for determination of outgassing rate q@sub out@ and total amount of released gases using capacitance manometer during pump down and SRG after the chamber was sealed-off. The initial q@sub out@ at room temperature after initial degassing was in the range of q@sub out@=10@super -12@ mbar l s@super -1@cm@super -2@ with a tendency of slight decrease with time. Thermal dependence of outgassing rate was determined from SRG measurements in the temperature range from 19 to 50 deg. C over several days with ability to extract the offset value by means of IG pump. The bake-out of the chamber was repeated for 72h at 200 deg. C which lead to the decrease of outgassing rate to one tenth of the initial value. The results show the benefit of using a moderately thin wall and agree well with a model of recombination limited hydrogen outgassing from stainless steel. @FootnoteText@ B.C.Moore: Atmospheric permeation of austenitic stainless steel, J.Vac.Sci.Technol., A16(5), 3114-3118, 1998 V.Nemanic,T.Bogataj: Outgassing of a thin wall stainless steel chamber, Vacuum, 50, 3-4, 431-437, 1998

9:00am **VT-ThM3 Review of Models for the Outgassing of Water Vapor from Metallic Surfaces**, ***B.B. Dayton,*** Consultant **INVITED**

The variation of outgassing rate with pumping time for water vapor from unbaked metal surfaces has been shown by several authors to depend on the ratio of pumping speed to exposed surface area, the time of initial exposure to the atmosphere at a known humidity, the ambient temperature, the thickness and porosity of the oxide layer on the metal surface, and the pretreatment or conditioning of the surface. The outgassing behavior differs radically from that for plastics and elastomers, and many authors have attempted to explain this behavior on the basis of theoretical models for the molecular kinetics. These models will be reviewed and evaluated. The present author will suggest a new model based on rapid physical adsorption and desorption in the oxide layer, with heat of adsorption in the neighborhood of 10 kcal/mol (corresponding to two hydrogen bonds), during atmospheric exposure followed by a slower (low probability) transition from a physisorbed precursor state (with activation energy of about 5 kcal/mol corresponding to breaking of one of the hydrogen bonds) to a "weak chemisorption state'', involving rotation and wobble about the remaining single hydrogen bond until an exposed pair of electrons associated with the water molecule has been captured in the positive field of a nearby metal ion without necessarily breaking the hydrogen bond to the oxide ion. Outgassing then involves the desorption of the undissociated water molecule from this bound state to the gas phase with an energy of desorption ranging from 20 to 30 kcal/mol.

9:40am **VT-ThM5 Measuring and Locating Internal Helium Leaks in the RHIC Insulating Vacuum System**, *R. Davis,* Brookhaven National Laboratory, U.S.; *C. De La Parra,* ***H.C. Hseuh****, P. Mickaliger, D.J. Pate,* Brookhaven National Laboratory

The Relativistic Heavy Ion Collider (RHIC) uses superconducting magnets to bend and focus the high-energy particle beams. Strings of these magnets are enclosed in insulating vacuum cryostats up to 480 meters long. The cryostat vacuum must be 10@super -5@ Torr or less to minimize heat transfer from the ambient cryostat wall to the 4K magnet cold mass. There are over 25,000 in-situ welds of internal helium lines with a total weld length over 7 km. Helium leaks greater than 10@super -5@ std.cc/sec (ambient leak rate) in these welds were located and repaired. The methods of locating these leaks in the long cryostat volumes will be described. Smaller leaks were monitored during the cool down of the magnets. The correlation of the leak rates with the helium line temperature and pressure will be presented.

10:00am **VT-ThM6 Contraflow Leak Detectors with Improved Sensitivity Under High Helium Background Conditions***, ***C. Dong,*** Old Dominion University; *G.R. Myneni,* Thomas Jefferson National Accelerator Facility; *G.A. Rooks,* Varian Vacuum Technologies

The leak detection sensitivity of the contraflow leak detectors is limited in general by its high operating pressure. Helium is known to be trapped in the fore pump of the leak detector and contributes to the large background signal particularly after leak checking large leaks (~1.0e-4 Pa. l/s). The trapped background helium can be effectively removed by purging the system with dry nitrogen. Such nitrogen purge is found to shorten the clean up period of leak detector by 90%. A novel method was developed to detect small leaks in large background helium signal. This method includes the lowering of pumping speed and adjustment of leak detector parameters such as the reduction of emission current and lowering the zero offset to minimize the background helium signal. We are able to leak check 3.0e-8 Pa. l/s and smaller leaks in a background signal of 4.0e-6 Pa. l/s of helium with 20 % accuracy. * This work was supported by U.S. DOE Contract No. DE-AC05-84ER40150 and Varian CRADA No. SURA-97-S002

10:20am **VT-ThM7 LIGO Beam Tube Component and Module Leak Testing**, ***W.A. Carpenter****, P.B. Shaw,* Chicago Bridge and Iron Co.; *R. Weiss,* Massachusetts Institute of Technology; *L. Jones,* California Institute of Technology

LIGO (Laser Interferometer Gravitational-wave Observatory) is a joint project of the California Institute of Technology and the Massachusetts Institute of Technology funded by the National Science Foundation. The project is designed to detect gravitational waves from astrophysical sources such as supernova and the formation of black holes. The LIGO project constructed facilities at two sites in the U.S. Each site includes two perpendicular laser beam tube lines (each 4 km long) which join at one end to form an "L" shape. The beam tube is a stainless steel, ultra high vacuum tube which will operate at a vacuum of 1 x10@super -9@ torr or better. The beam tube was manufactured using a custom spiral weld tube mill and was manufactured with special emphasis on reduced outgassing rates. The integrity of the beam tube was assured by leak testing each component of the beam tube system prior to installation and then to leak test each 2 kilometer isolatable beam tube module after completion. This paper discusses the leak detection procedures used to leak test 16 km of 1.25 m diameter UHV beam tube used in the LIGO project. The beam tube was leak tested in four steps including leak testing fabricated tube sections, local leak testing of 250 mm diameter valve and valve nozzles, leak testing circumferential welds joining tube sections together and final leak testing of the installed beam tube module. The component leak testing included 800 tube sections which were 20 m long, 808 circumferential welds and 72 valved nozzles. Each component was tested to a sensitivity of better than 1x10@super -10@ atm. cc/sec of helium. The leak test of the 2 kilometer beam tube module would have been extremely difficult and expensive using standard helium leak detection techniques. Therefore, a method was developed utilizing a Residual Gas Analyzer (RGA) to measure the leak tightness of the two kilometer long modules. A method was also developed to utilize nine RGAs to locate any detected leaks.

10:40am **VT-ThM8 Low-flow Measurements Techniques for Calibrating Mass Flow Controllers*, S.A. Tison,*** Millipore Corporation, U.S.A*; C. Ruppert,* Millipore Corporation

Many semiconductor processes require stable and known flow of a gas or multiple gases. Flows ranging from 100 slpm (7 x 10-2 mol/s) to 10 sccm (7 x 10-6 mol/s) are common. Primary standard flowmeters have been developed to measure these flows and are in common use.. The most common techniques include gravimetric and those based on volume displacement at constant pressure. Recently a significant number of processes are using much lower flows with flows as low as 0.1 sccm (7 x 10-8 mol/s) being required. Most of the traditional techniques for measuring gas flow are not well suited for measurements in this range. Becasue of this, process tuning and reproducability can be adversely affected by errors in the mass flow controllers which are induced by their calibration. Two of the most common instruments used for low-flow measurements , a constant volume flowmeter and a laminar flowmeter, were compared with a number of gases including nitrogen, hydrogen, and sulfur hexafluoride. The agreement of the techniques was generally within 0.5% over a range of 1 sccm (7 x 10-7 mol/s) to 10 sccm (7 x 10-6 mol/s) and somewhat worse for lower flows. The comparison data and attributes of these two types of standards is discussed.

11:00am **VT-ThM9 Dimensionless Parameters for Laminar Flowmeters*, R.F. Berg,*** National Institute of Standards and Technology

Laminar flowmeters have strong advantages as transfer standards for measuring low flow rates of gases. Ideally, the difference between the flowmeter's entrance and exit pressures, P@sub 1@ and P@sub 2@, is that associated with viscous, creeping flow. One can then approximate the volume flow rate by the value Q@sub 0@ = @pi@R@super 4@(P@sub 1@-P@sub 2@)/(8@eta@L). This is the Hagen-Poiseuille relation which describes an incompressible fluid of viscosity @eta@ flowing through a capillary of circular cross-section with length L and radius R. In practice, the actual flow rate Q is described by the discharge coefficient C@sub d@=Q/Q@sub 0@. Achieving an accuracy of 0.1% requires a series of corrections to C@sub d@, each associated with at least one dimensionless parameter. Identifying the form of these corrections allows one to define useful dimensionless parameters. Important deviations of C@sub d@ from unity occur because the gas is compressible. A steady mass flow rate causes the volume flow rate to depend on position along the capillary. This corrects C@sub d@ by a factor proportional to (P@sub 1@+P@sub 2@). Furthermore, the additional pressure drop associated with the expanding gas adds to C@sub d@ a term proportional to the Reynolds number Re. Recent analysis of capillary viscometers by van den Berg and coworkers showed that the nonparabolic flow associated with the expanding gas increased the size of this term. Their result is used here to describe the performance of a laminar flowmeter. Dimensionless parameters incorporated in this correction include Re, the pressure ratio P@sub 2@/P@sub 1@, and the aspect ratio R/L. Other significant dimensionless parameters include the Mach number, the ratio of the gas's slip length to the capillary's radius, the Dean number characterizing centrifugal effects in a coiled capillary, and the correction terms in the gas's virial equation of state.

11:40am **VT-ThM11 Decay Rate of Photoresist Outgassing from Ion Implantation*, A.S. Perel,*** *T.N. Horsky,* Eaton Corporation

Outgassing of hydrocarbons results from ion implantation into wafers covered with photoresist. Depending on the implant energy and current, the quantity of gas can be large and therefore high effective pumping speeds are important to minimize interference with the ion beam. We measured the time dependence of this outgassing to determine the rate that the outgassing decays. This information is relevant for design of implanter process chambers with rotating disks. The photoresist outgassing decay rate determines the geometry of the gas load, and can be used to determine pump placement and chamber design. The experiment involved a single photoresist wafer and 12 dummy wafers on a 13 pad disk. By measuring the collector current directly from the ion gauges we were able to time-resolve pressure measurements to better than 1 ms. Two pressure pulses were measured, the pulse that results from the implant and a pulse that results from the outgassing wafer passing the gauge. The relative difference in amplitude and time, as observed from two gauges, were used to calculate the outgassing decay rate. We reduce several data sets collected at different disk rotational speeds to fit a candidate model for the time-dependence and mechanism of photoresist outgassing.

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