

Tuesday Afternoon, November 5, 2002

Vacuum Technology

Room: C-104 - Session VT-TuA

Vacuum System Architecture and Specialized Analytical Techniques

Moderator: J.L. Provo, Sandia National Laboratories

2:00pm **VT-TuA1 Present Status of the KEKB Vacuum System.** K. Kanazawa, S. Kato, Y. Suetsugu, H. Hisamatsu, M. Shimamoto, M. Shirai, High Energy Accelerator Research Organization (KEK), Japan

The KEK B-Factor (KEKB) is an electron-positron collider with an asymmetric energies consisting of two rings, that is, the High Energy Ring (HER) for 8.0 GeV electrons and the Low Energy Ring (LER) for 3.5 GeV positrons. The design beam currents are 1.1 A and 2.6 A with 5120 bunches for HER and LER, respectively. Each ring has a circumference of 3016 m and most of beam chambers are made of oxygen free copper for its ability to withstand the intense heat load and to shield effectively the radiation from high beam currents. The pumping scheme is a combination of Non-Evaporable Getter (NEG) pumps and sputter ion pumps. The commissioning of KEKB started in December 1998. At the end of March 2002, the achieved stored currents were about 0.93 A and 1.44 A for HER and LER, respectively, with 1200 bunches. The KEKB is now able to serve the world-record luminosity of $7.2 \text{ nb}^{-1} \text{ s}^{-1}$ for the BELLE detector. The vacuum system has been operating satisfactorily. The average pressure of about $1 \times 10^{-7} \text{ Pa}$ is achieved now for both rings during the operation. The coefficient of the photon stimulated gas desorption (PSD) decreased steadily to almost 1×10^{-6} molecules photon^{-1} at the integrated linear photon flux of about 3×10^{25} photons m^{-1} . Most of vacuum components, such as Helicoflex sealing (Le Carbone Co. Ltd.) and the vacuum bellows with RF-shield structure, have been working well. The severest trouble had been the beam-induced troubles of the movable masks, but the newly developed masks were installed and are now in use without serious problem. One of the latest issues is the excess heating of bellows, chambers or pumps coming from the electromagnetic field excited by the intense bunched beam. A non-linear dependence of pressure on beam current has been observed in LER. That seems to deeply relate to the electron multipactoring, which causes the electron cloud instability. Here we will summarize the experiences and the present status of KEKB vacuum system, and touch briefly the future plan.

2:20pm **VT-TuA2 Development of Sputtering System for Large-Area Deposition of $\text{CuIn}_{1-x}\text{Ga}_x\text{Se}_{1-y}\text{S}_y$ Thin-Film Solar Cells.** N.G. Dhere, A.H. Jahagirdar, A.A. Kadam, V.S. Gade, H.P. Patil, University of Central Florida

Manufacturing cost of $\text{CuIn}_{1-x}\text{Ga}_x\text{Se}_{1-y}\text{S}_y$ (CIGS) thin-film modules is expected to become cheaper than that of crystalline silicon modules within 5 years. At present, commissioning and reaching full production of thin film modules is delayed because of the non-availability of turnkey manufacturing plants. Few university laboratories are conducting research on design and construction of PV plants. CIGS thin-film solar cells are being prepared routinely at FSEC on glass and metallic foil substrates for terrestrial and space applications. Earlier the size was limited to $3 \times 3 \text{ cm}^2$. This paper presents results of development efforts in design and construction of large-area sputtering system for large-area ($15 \times 15 \text{ cm}^2$) CIGS thin-film solar cells. The system has the potential of serving as a nucleus of a pilot plant for fabrication of CIGS minimodules. It could be used for simulating full-scale production set-up. Stepper-motor controlled, linear substrate movement and uniform argon distribution set-ups were designed and built. RF tuning network was modified for optimum impedance matching. Initial problems of bowing of the backing diaphragm, possible formation of air pockets, restriction of effective water flow and consequent heating of the target material were resolved by increasing the thickness of the backing plate and redesigning the structural members. Thickness distribution was optimized by modifying the magnetic field distribution in the middle 15-cm portion of the $10 \times 30 \text{ cm}^2$ magnetron sputtering sources by selectively removing nickel-coated soft-iron pieces at the rear. The present optimum configuration has resulted in thickness variation of $\pm 3\%$ over $11.5 \times 10 \text{ cm}^2$ for Mo, CuGa, In, ZnO, and ZnO:Al layers. Magnetic field is being boosted at extremities to avoid precipitous $\sim 15\%$ drop beyond 11.5 cm and to achieve thickness uniformity of better than $\pm 2\%$ over $12.7 \times 12.7 \text{ cm}^2$ and $\pm 3\%$ over $15.3 \times 15.3 \text{ cm}^2$ areas.

2:40pm **VT-TuA3 Ultra-sensitive Detection of Helium Release from Metal Tritides.** J. Poths, T.J. Venhaus, F.J. Steinkruger, Los Alamos National Laboratory

INVITED

Static noble gas mass spectrometry has been in use for ultra-sensitive analysis of samples in the geological community for almost 5 decades. We have been using a magnetic sector instrument created by Al Nier at the University of Minnesota in both geological and nuclear research. In static mode of operation, the sample is equilibrated into the volume of the instrument with no pumping, rather than flowing through the source and into a pump as in a standard dynamic mass spectrometry. Thus, static operation provides a factor of 10,000 increase in sensitivity. We have recently applied this technique to analyzing the release of helium-3 from erbium ditritide films. The increased sensitivity has allowed us to compare helium release on timescales ranging from 5 minutes to 100 days. We confirm that helium release is highest at the beginning of an erbium ditritide film's lifetime, then decreases after a few months to a steady rate. Interestingly, during this period of steady-state release, the helium release rate seems to be independent of the buildup of helium-3 levels in the film. This observation suggests that at the moment of tritium decay a helium-3 atom is either immediately released or is trapped and no longer accessible for release. At steady-state, the ratio of helium released to helium decayed during storage is about 0.008.

3:20pm **VT-TuA5 Quantification of Mass Spectra in Experiments with Deuterium.** B. Zajec, V. Nemanic, M. Zumer, Institute of Surface Engineering and Optoelectronics, Slovenia

For monitoring the interaction of gaseous hydrogen with surfaces or bulk materials, deuterium is frequently applied as a tracer gas for the quadrupole mass spectrometer (QMS) analysis. In fast and steady processes, the major peak at mass number 4 is frequently a good indicator of deuterium involved reactions while peaks at mass numbers 2 and 3 may be neglected. In slow and transient processes, the weak peaks at mass numbers 2, 3 and 4 must be considered for a quantitative analysis. Tracing reactions where deuterium molecules dissociate is troublesome since the mass number 2 overlaps with the hydrogen background. The problem to eliminate the QMS artefacts is usually solved by calibration of the instrument with a suitable low deuterium flux, but any further resolution of the background hydrogen originated from the chamber surfaces is very inaccurate. We present the results of a calibration procedure of two QMS mounted on a well outgassed UHV system, realised in a specific way. Each of the QMS was pumped in line but could be also separated from the system by a valve. A constant deuterium or hydrogen inflow was set from $10^{-8} \text{ mbar l s}^{-1}$ to $10^{-6} \text{ mbar l s}^{-1}$ by observing the pressure rise in the chamber by means of a calibrated capacitance manometer. The calibration of both QMS was taken in the dynamic mode for both gases. Further on, the spectra of deuterium accumulated in the UHV system for a defined period of time were compared to the calibration spectra. From the difference, we could resolve the extent of mass numbers 2 and 3 produced in the QMS from the contribution of the deuterium participated in surface reactions at the chamber wall. It was thus shown that after admittance of pure deuterium into the UHV chamber at 10^{-4} mbar , its exchange with the adsorbed hydrogen led in some hours to a noticeable changed proportion of mass numbers 2, 3 and 4. This could not be predicted from the low background outgassing rate.

3:40pm **VT-TuA6 Mass Spectrometric Determination of Hydrogen and Hydrogen Isotopes from Thin Films.** J.F. Browning, Sandia National Laboratories

The absolute measurement of hydrogen and hydrogen isotope concentrations in materials is of interest to many areas of both applied and fundamental research. In this work we describe a technique for the high accuracy determination of hydrogen concentration in thin films. The technique involves the thermal desorption of hydrogen from the film at a temperature of 900°C . Application of the ideal gas law to such experiments is questionable at best due to the extreme temperature range existing between the thermal desorption system and the mass spectrometer inlet system. To overcome issues associated with such extremes in temperature we use a response function technique to quantitate the total molar quantity of gas evolved from the film followed by high resolution mass spectrometric determination of the constituent components. Molar concentrations in the range 10^{-8} to 10^{-5} are routinely determined to an overall uncertainty of $\pm 1\%$.

4:00pm **VT-TuA7 Quantification of Gas Load from Ultra-small GDP Capsules, S.D. Balsley**, Sandia National Laboratories

A-priori knowledge of the gas composition of 10^{-8} liter capsules manufactured by General Atomics Corporation (GA) and used in inertial confinement fusion (ICF) experiments at Sandia National Laboratories' Z-pinch facility is important to experimentalists and modelers. These Z-pinch driven capsules are larger than any ever fielded by any other ICF program, and as such are part of an ongoing research program at GA and Sandia. Total pressure determinations via burst tests, although useful, lack information regarding the composition of gas mixtures. Here we present a novel technique for quantitatively determining partial pressures of gas loaded capsules whereby a low-background vacuum fixture for capsule bursting is coupled with a Finnigan-MAT 271 mass spectrometer. Typically, capsules consist of a 50 μ m thick polymer coated with a 3 μ m polyvinyl alcohol layer. Capsules are diffusion filled with a deuterium-argon mixture that is pre-defined by model calculations. Nominal fill pressures for deuterium-argon capsules are 17atm and 0.075atm, respectively. Other gas fills are also used, including deuterated methane (CD₄) mixed with minor quantities of argon or tetramethylsilane. Initial test results from several argon filled capsules agree well with stated fill pressures. Subsequent analysis of sister capsules similar to those imploded at Z-pinch in 2002 show good correlation between stated fill quantities and expected deuterium loss due to diffusion. It is envisioned that application of this technique will be important for gas fill validation of target capsules to be used in the National Ignition Facility, scheduled to begin testing in 2003-2004.

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4:20pm **VT-TuA8 How to Have Clean Surfaces in an Unclean World, R. Robinson, D.D. Allred, A. Guillermo, R. Sandberg, A Jackson**, Brigham Young University, *M.K. Newey*, University of Maryland

Clean surfaces that are exposed to the atmosphere rapidly become dirty. Even though a surface may be carefully prepared and well characterized in its preparation or deposition environment, there is no guarantee that it will remain unchanged minutes after it is exposed to the air. We will report our measurements on the amount of contamination a sample acquires from various activities including handling with various kinds of gloves, sitting face up or down in the lab for several hours, storage in wafer carriers etc. We will also discuss the use of several cleaning techniques: UV light+ozone, plasma ashing, and polymer (Opticlean) for removing dust and/or the contamination layers in preparing materials for EUV/VUV (8-110 eV) and/or AFM measurements. Experience from silicon-based microelectronics is instructive but not definitive here. Answers depend in part on the identity of the surface. XPS shows the presence of carbon and oxygen in most contamination layers. Spectroscopic ellipsometry's advantages include: it can be used in air, measurements are rapid and it is sensitive to the relative thickness of layers even in the sub nm range. Measurement is the first step to understanding which can lead to control. We are making materials for VUV and EUV (8-100 eV) optical applications where the presence of a few nanometers of carbon and/or oxygen containing materials can have a remarkable effect on the performance of the surface. A companion group is doing AFM. Here the presence of organic contamination can clog the tip. We will report our answers to questions such as: How fast does a surface acquire its accidental carbon-containing overcoat? What can be done to block or ameliorate deterioration? Can the sample be handled, cut and stored for several hours before the next measurements? How can a sample be restored or recleaned without losing its essential characteristics? When does storage introduce its own problems?

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