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by

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GAS DESORPTION AND SURFACE CONDITIONING OF A SYNCHROTRON RADIATION SOURCE

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ABSTRACT

Synchrotron radiation induced neutral gas desorption from baked Al alloy vacuum chambers cleaned using alkaline detergents has been measured in a test beam line at the DCI storage ring at LURE, Orsay, France.

The gases desorbed were H_2 , CH_4 , CO and CO_2 and with time the H_2 and CO desorption decreased as $D^{-1/2}$ where D is the radiation beam dose in mA hours. Similar results for electron induced neutral gas desorption from small samples showed a similar behaviour and led to the proposal of a model of gas diffusion from near surface layers and from the bulk to the surface which determined the desorption characteristics. For H_2 the model described well the behaviour of the H_2 desorption.

Measurements of the photoelectron currents produced at 11 mrad glancing angle of incidence and also at normal incidence, when compared with calculation, showed differences which could be explained if the photons with energies up to between 1.2 and 1.5 keV were reflected at 11 mrad with reflectivities approaching 1. Using published values of the complex refractive index for aluminium oxide high reflectivities up to 1 keV were calculated.

In a special test chamber equipped with sets of photoelectron probes it was established that, at glancing angles of incidence of 11, 20 and 40 mrad, a substantial - up to 20% - fraction of the synchrotron radiation is scattered around the vacuum chamber from the initial point of impact.

INTRODUCTION

Synchrotron radiation induced gas desorption is one of the most important effects which limit the performance of synchrotron light sources. This dynamic desorption is, unfortunately, not directly related to the static vacuum achieved in the laboratory. Low ultimate pressures in no way imply that the surface is devoid of gas. It only means that the binding energies of the remaining adsorbed surface gases after bakeout are high enough to give a low thermal outgassing rate at room temperature and that the pumping speed is high enough to maintain this low pressure.

In operation, synchrotron light sources are therefore, due to the gas desorption from the walls, self cleaning but the time dependence of this process and the factors influencing it are not well understood. It is the purpose of this publication to present the

results of a series of measurements of synchrotron radiation induced neutral gas desorption from test vacuum chambers installed in a dedicated beam line on the DCI positron storage ring at the LURE laboratory, Orsay, France and of electron induced gas desorption from small samples in the laboratory in order to have a better understanding of the mechanisms involved.

CLEANING

It has been shown that Al vacuum chambers are best cleaned by etching in alkaline solutions¹. The present test chambers were part of the series production of vacuum chambers for the CERN LEP 100 GeV e^+e^- storage ring and had been cleaned by a high pressure jet process using alkaline fluids. An alkaline detergent (Almecco 29) with a removal rate at 2% concentration and 60°C, of about 1100 Å per minute was sprayed inside and outside with high pressure jets (~ 100 bar). In addition a second product (Amklene D Forte) diluted to 2% with a removal rate 6 times that of Almecco 29 at 60°C was also sprayed into the vacuum chamber.

Rinsing with demineralized water was also carried out with the same system. Drying was by hot air at ~ 80°C.

PHOTON INDUCED GAS DESORPTION

The experimental set-up is shown in Fig. 1 and has been described fully in a previous publication².

Fig. 1. The experimental set-up

In a clean, baked, all-metal ultra-high vacuum system with a pressure in, for example, the 10^{-11} Torr range, the residual gas is usually composed mainly of H_2 . A typical spectrum is shown in Figure 2a, the composition of which is H_2 (82%) with traces of CH_4 (4%), CO (9.5%) and CO_2 (4.5%).

The composition of the residual gas is, of course, a reflection of the nature of the gas species adsorbed on the surface but gives little indication of the quantity since, as stated previously,

Fig. 2. The residual gas spectrum before (a) and during (b) exposure to synchrotron radiation.

where the pressure has risen from 7×10^{-11} Torr to 5×10^{-7} Torr is shown in Figure 2b. There it is seen that the composition is now 31% H_2 , 7% CH_4 , 24% CO and 38% CO_2 , i.e. apart from H_2 the principal contaminating gases in the vacuum system are all C based.

In Fig. 3 are shown the dependence of the absolute partial and total pressures per mA of beam as a function of the beam dose in a

Fig. 3. The dependence of the total and partial pressures as a function of beam dose.

molecules in high binding energy states desorb little at room temperature.

Under energetic particle bombardment the gases desorbed are H_2 , CH_4 , CO and CO_2 . Although visible in the residual gas H_2O does not appreciably change in the residual gas spectrum.

The pressure in the vacuum system may rise by a factor of 10^3 and have a composition drastically different from the initial.

A typical spectrum from a vacuum system subjected to synchrotron radiation

where the pressure has risen from 7×10^{-11} Torr to 5×10^{-7} Torr is shown in Figure 2b. There it is seen that the composition is now 31% H_2 , 7% CH_4 , 24% CO and 38% CO_2 , i.e. apart from H_2 the principal contaminating gases in the vacuum system are all C based.

Expressed in terms of the specific absolute pressure rise (Torr mA⁻¹) the first exposure to synchrotron radiation shows in order of importance CO_2 , H_2 , CO and CH_4 . The partial pressure of H_2O in the baked system is low and

increases during photon bombardment insignificantly.

At the maximum beam dose of 1.6 A hours H_2 is the leading component followed by CO while CO_2 and also CH_4 show a relatively steep and effective clean-up behaviour.

The H_2 clean-up rate, in particular, varies as $Dose^{-\frac{1}{2}}$.

Since the magnitude of the partial pressures depends on the actual pumping speed for each gas in our system we have expressed the data of Fig. 3 in the form of a molecular desorption yield (molecules per incident photon) where the actual pumping speeds have been taken into account.

As can be seen from Fig. 4, in terms of this molecular desorption yield H_2 becomes the leading gas while the order of the other gases remains unaltered.

Fig. 4. The desorption coefficients as a function of the photon dose.

PHOTOELECTRON PRODUCTION

It has been proposed that the desorption of neutral gas from surfaces subjected to bombardment by synchrotron radiation takes place mainly due to photoelectrons,^{3,4,5,6}; however, direct desorption by the photons is not excluded but the two effects will occur simultaneously and are difficult to separate. In an effort to shed some light on the desorbing mechanism we have placed in the central part of the chamber a 20 cm long, 1 mm diameter stainless steel wire suspended from an insulated feedthrough, along the axis of the test chamber with which photoelectrons produced in the vicinity may be collected and measured by applying a positive bias to the wire. Thus simultaneously the photocurrents inside an Al vacuum chamber and the photon induced gas desorption could be measured as a function of the radiation spectrum i.e. beam energy.

Fig. 5. The measured photoelectron current on the wire as a function of the beam energy.

Fig. 6. The specific total pressure increase as a function of beam energy.

A first measurement of the photoelectron currents and the gas desorption as a function of beam energy and at 11 mrad glancing angle of incidence was made before any beam cleaning and a second measurement after a beam cleaning does of ~ 600 mA hours.

The results are shown in Fig. 5 and Fig. 6 for the photoelectron currents and total gas desorption respectively. Since the stored beam current decreases with time both the photocurrents and the pressure are divided by the beam current in mA

The total specific pressure rise and the collected photoelectron current both follow a similar steep rise with stored beam energy. Continued exposure to the synchrotron radiation cleans the vacuum chamber and results in a corresponding reduction of the pressure rise which is, however, more pronounced at higher beam energy.

At the downstream end of the test chamber a flat Al alloy plate was used to measure the normal incidence photoelectron current. This electrode was mounted on a UHV high voltage feedthrough and could be biased up to 1 kV.

Fig. 7. The measured and calculated photoelectron current from the end plate as a function of beam energy.

trons radiation while the second set (after beam cleaning) was taken after a prolonged exposure to synchrotron radiation at 1.1 GeV reflected from the side wall where it was incident at 11 mrad.

Comparing the dependence of the photoelectron currents with beam energy for normal incidence (Fig. 7) and 11 mrad glancing angle of incidence (Fig. 5) it can be seen that the form of the two appear very different.

PHOTOELECTRON CURRENT CALCULATION

The number of photons in the synchrotron radiation beam per second, dt , per mA, di , per eV, $d\epsilon$, and per radian of beam orbit, da , is given by

$$\frac{d^4 N}{d\epsilon dt di da} = \frac{6.95 \times 10^3}{2\pi} \frac{\rho}{E^2} \int_y^\infty K_{5/3}(x) dx \quad (1)$$

where E is the beam energy in GeV

ϵ is the photon energy in eV

$$\epsilon_c \text{ is the critical energy} = \frac{2.2 \times 10^3 E^3}{\rho}$$

is the bending radius in metres = 3.82 m for DCI

$$y = \epsilon / \epsilon_c \quad \text{and}$$

$K_{5/3}$ is a modified Bessel function of second kind of order 5/3.

With the test chamber in the straight through position, the photons incident on the Al plate and a bias of - 150 V a saturated photoelectron current could be obtained at all beam energies.

The results of these measurements which cover the beam energy from 1.1 GeV to 1.8 GeV - corresponding to a critical energy of the photon spectrum between 773 eV and 3.38 keV - are shown in Fig. 7.

Of the two sets of experimental data the first (before beam cleaning) refers to the initial measurement at the first exposure to synchro-

The total number of photoelectrons produced by the synchrotron radiation beam is obtained by integrating the product of the spectrum given in (1) and the normal incidence photoelectric yield Y for Al oxide⁷.

For normal incidence the calculated photoelectron current is shown in Fig. 7 and there it is seen that the agreement with the measured values is to within 15%.

At glancing angle of incidence good agreement between the calculated and measured values of the photoelectron current are only obtained (Fig. 5) if the effect of reflection of the photons is taken into account⁸.

If one assumes that at grazing incidence a non-negligible part of the low energy photon spectrum is reflected, these photons will contribute neither to the photoelectron production nor to the gas desorption.

Fig. 8. The calculated reflectivity of photons on Al oxide at 11 mrad grazing angle of incidence

published values of the real and imaginary parts of the refractive index of aluminium oxide¹⁰.

Introducing this assumption of high reflectivity up to a certain photon energy into the photoelectron current calculation yields good agreement with the experiment if a reflectivity of 1 for photons up to between 1.2 and 1.5 keV is assumed⁹. Such a high reflectivity for photons of these energies at grazing angles of incidence is not unrealistic as may be seen from Fig. 8. This figure shows the high reflectivity, up to about 1 keV, for 11 mrad angle of incidence and has been calculated using

ELECTRON INDUCED GAS DESORPTION

The apparatus has been fully described in reference 11 and here we shall present only some results of the gases desorbed and the clean-up rate when bombarding Al samples continually with 500 eV electrons.

After a 24 hour 150°C bakeout the gases desorbed by the 500 eV electrons (molecules per electron) are in order of importance H_2 , CO, CO_2 and CH_4 , i.e. the same as for the photon induced desorption.

In Fig. 9 is shown the dependence of the molecular desorption on the electron dose and the similarity to Fig. 6 is evident. For H_2 and CO the desorption decreases with time t as $t^{-1/2}$ (at constant bombarding current the dose is directly proportional to time).

A model describing the clean up of the surface as a process where the time dependence is determined by gas transport via diffusion to the surface has been proposed¹². The model is restricted to the behaviour of H_2 because, for H_2 , the effect of chemical reactions may be neglected and fundamental data such as the concentration in the bulk are well known.

However, in all our measurements CO shows a similar behaviour to H_2 (CO_2 and CH_4 are somewhat different) thus the proposed model may also be valid for CO.

In Fig. 9 the full line shows the calculated clean-up rate for H_2 using as parameters the diffusion coefficient for H_2 and the concentration of H_2 in bulk Al. It can be seen that the calculated curve agrees well with the measured data.

Fig. 9. The measured and calculated electron induced desorption coefficients as a function of electron dose.

SCATTERED RADIATION

Synchrotron radiation is emitted in a narrow cone in the plane of the orbit and is incident on a narrow strip of vacuum chamber wall typically some mm in height. At this initial point of impact photoelectrons are produced which, depending on whether a magnetic field is present or not, may return close to their point of origin or traverse the vacuum chamber and desorb gas from there. In addition some photons may be reflected and/or scattered and thus also cause desorption of gas for other parts of the vacuum chamber not directly hit by synchrotron radiation.

To obtain a better knowledge of the importance of the scattered radiation with our geometry and photon spectrum (critical energy) a test chamber equipped with two sets of photoelectron probes as shown in Fig. 10 was installed in the synchrotron radiation beam line. A detailed drawing of the electrode ports, the construction of the individual electrodes and the synchrotron radiation beam dimensions and shape at these positions are also shown. After installation the complete system was baked at 150°C for 24 hours.

Fig. 10. The special test chamber with its photoelectron probes

A typical result at 11 mrad glancing angle of incidence with the first set of probes is shown in Fig. 11. From this figure and from analysis of the other results obtained it is evident that the directly illuminated probe always gives the largest photoelectron signal. The indirectly exposed probes, however, show a nearly uniform photocurrent implying that the photons are reflected diffusely rather than specularly.

Fig. 11. The photoelectron currents measured on the first set of 5 electrodes at 11 mrad glancing angle of incidence for various beam energies.

It must be remembered that the measurements are made with a negative bias on the electrodes thus what is detected is the photoelectron current due to incident photons - secondary electrons and primary photoelectrons from the point of impact of the synchrotron radiation are not seen.

Fig. 12. The fraction of the total photoelectron current due to scattered photons as a function of beam energy.

ratio increases steeply with beam energy and exceeds 20% at the maximum beam energy of 1.8 GeV. As the angle of incidence increases the locally measured fraction of scattered radiation increases and also a considerable number of photons appears to be scattered in the forward direction along the test chamber.

Based on a series of measurements, the fraction of the total photoelectron current due to scattered photons has been estimated with some simplifying assumptions about the uniformity of the polar distributions. The results are shown in Fig. 12 which gives, as a function of beam energy, the ratio of the photoelectron currents for the indirectly and directly exposed parts of the chamber. It can be seen that this

CONCLUSIONS

The fact that relatively large photoelectron currents (several μA per mA of beam) are measured in the test chamber when exposed to synchrotron radiation and that the form of the beam energy dependence of the pressure increase is similar to that of the photoelectron currents makes it tempting to assume that the photoelectrons are predominantly responsible for the gas desorption.

The similarity between the gases desorbed by synchrotron radiation and electrons, i.e. H_2 , CH_4 , CO and CO_2 , and the form of the clean-up curves also support the view that the photoelectrons produce much of the gas desorption.

A model of gas diffusion from near surface layers and from the bulk to the surface was proposed which determined the desorption characteristics. For H_2 this model described well the behaviour of the H_2 desorption with time. A similar behaviour was observed for CO thus the model may also be true for this gas.

Measurements of the photoelectron currents produced at 11 mrad glancing angle of incidence and also at normal incidence, when compared with calculation, showed differences which could be explained if the photons with energies up to between 1.2 and 1.5 keV were reflected at 11 mrad with reflectivities approaching 1. Using published values of the complex refractive index for aluminium oxide high reflectivities up to 1 keV were calculated.

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