EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH

CERN - AT DIVISION

CERN/AT-VA/90-21

COMPARISON OF SYNCHROTRON RADIATION INDUCED GAS DESORPTION FROM Al, STAINLESS STEEL AND Cu CHAMBERS

A. G. Mathewson, O. Gröbner, P. Strubin
P. Marin *), R. Souchet *)

Abstract

The synchrotron radiation induced neutral gas desorption coefficients for Al, stainless steel and Cu have been measured by exposing test vacuum chambers made from those materials to 2.95 keV critical energy synchrotron radiation in a dedicated beam line on the DCI positron storage ring at the LURE Laboratory, Orsay, France.

In addition to the initial desorption coefficients, the decrease of the gas desorption with photon dose is also described. Al was found to have the largest initial desorption coefficients, with Cu a factor of about 10 lower and the lowest desorption coefficients were from stainless steel, where a 950°C vacuum degassing treatment had little effect.

Integration of the desorption coefficients with respect to photon dose gave the total quantity of gas desorbed. For a photon dose of $3 \cdot 10^{21}$ photons/m, almost 1 Torr l/m of H_2 , CH_4 , CO and CO_2 were desorbed from Al, 10^{-1} Torr l/m from Cu and $5 \cdot 10^{-2}$ Torr l/m from stainless steel.

In parallel, measurements of the photoelectron currents, believed to be the primary cause of the neutral gas desorption, were carried out. The highest photoelectron currents were measured in the Al chamber, followed by Cu with the lowest in the stainless steel chambers.

Using the results from the Al test chamber, the predicted pressure increases in the LEP vacuum system, when exposed to synchrotron radiation, agreed well with those actually measured.

*Invited paper presented at
the Vacuum Design of Synchrotron Light Sources Conference,
Argonne National Laboratory, 13-15 November, 1990*

Geneva, November 1990

*) P. Marin, R. Souchet, L.U.R.E., Orsay, France

COMPARISON OF SYNCHROTRON RADIATION INDUCED GAS DESORPTION FROM Al, STAINLESS STEEL AND Cu CHAMBERS

A. G. Mathewson, O. Gröbner, P. Strubin, C.E.R.N., Geneva, Switzerland
P. Marin, R. Souchet, L.U.R.E., Orsay, France

ABSTRACT

The synchrotron radiation induced neutral gas desorption coefficients for Al, stainless steel and Cu have been measured by exposing test vacuum chambers made from those materials to 2.95 keV critical energy synchrotron radiation in a dedicated beam line on the DCI positron storage ring at the LURE Laboratory, Orsay, France.

In addition to the initial desorption coefficients, the decrease of the gas desorption with photon dose is also described. Al was found to have the largest initial desorption coefficients, with Cu a factor of about 10 lower and the lowest desorption coefficients were from stainless steel, where a 950°C vacuum degassing treatment had little effect.

Integration of the desorption coefficients with respect to photon dose gave the total quantity of gas desorbed. For a photon dose of $3 \cdot 10^{21}$ photons/m, almost 1 Torr l/m of H₂, CH₄, CO and CO₂ were desorbed from Al, 10^{-1} Torr l/m from Cu and $5 \cdot 10^{-2}$ Torr l/m from stainless steel.

In parallel, measurements of the photoelectron currents, believed to be the primary cause of the neutral gas desorption, were carried out. The highest photoelectron currents were measured in the Al chamber, followed by Cu with the lowest in the stainless steel chambers.

Using the results from the Al test chamber, the predicted pressure increases in the LEP vacuum system, when exposed to synchrotron radiation, agreed well with those actually measured.

INTRODUCTION

In synchrotron radiation light sources and high energy electron or positron storage rings the vacuum chambers are usually made from Al, stainless steel or Cu. The choice of the material is usually determined by cost, fabrication complexity and heat load from the synchrotron radiation. Because of its high thermal conductivity and ease of brazing, copper is also widely used as an absorber of synchrotron radiation. With the proper chemical cleaning and careful handling, the static thermal outgassing from these three metals is very similar. However, when subjected to synchrotron radiation, the photon induced neutral gas desorption can exceed the thermal outgassing by several orders of magnitude and it is this effect which determines the vacuum

performance of the machine and decides the installed pumping capacity. With continued exposure to synchrotron radiation, the gas desorption decreases, i.e. the surface cleans up.

The initial photon induced gas desorption is important since it is this which determines the beam gas lifetime when the machine is started for the first time. The rate at which this desorption decreases with machine running time is more important since this determines the time for which the machine must be operated before acceptable beam-gas lifetimes can be obtained. Also the total quantity of gas desorbed is an important figure since it has consequences for the choice of the installed pumping capacity.

To obtain reliable figures for the desorption coefficients, the total amount of desorbed gas and the clean-up time for the vacuum system of the 27 km, 55 GeV electron positron storage ring LEP, test vacuum chambers made from Al, stainless steel and Cu were exposed to synchrotron radiation in a dedicated photon beam line on the DCI positron storage ring at the LURE Laboratory in Orsay, Paris¹. In addition, measurements of the photoelectron currents produced by the photons in these chambers were made.

EXPERIMENTAL DETAILS

A. Vacuum system

Synchrotron radiation of 2.95 keV critical energy from a bending magnet of the DCI storage ring is extracted from the machine and passes through a collimator which limits the angular divergence of the radiation to 4.7 mrad in both the horizontal and vertical directions. This vertical collimation attenuates the continuous photon spectrum below about 6 eV. The test chambers could be pivoted in the horizontal plane so that the photons were incident on the side wall of the chamber at 11 mrad glancing angle of incidence where 3.12 m were irradiated.

The test chambers were pumped via a 72.5 l/s conductance for N₂ by a combination of a 400 l/s ion pump and two Ti sublimation pumps of about 1000 l/s each.

Pressure measurements were made with calibrated Bayard-Alpert gauges and a quadrupole gas analyser.

Data taking and analysis were performed by a specially developed minicomputer system.

B. Test chamber treatment

The aluminium test chamber was elliptical in cross section (131 mm x 70 mm) and manufactured from extruded tube. The material is a precipitation hardenable alloy of type ISO Al Mg Si 0.5. The chamber

was chemically cleaned by spraying with an alkaline detergent at 60°C and about 100 bar, rinsed with cold demineralized water and dried with filtered hot air at 90°C².

The copper chamber was manufactured from 3 mm thick OFHC copper plate rolled in four 90 cm sections and electron beam welded to form a tube 131 mm in diameter and 3.6 m long. ConFlat flanges were vacuum brazed to each end. Before welding, each 90 cm section was chemically cleaned by first degreasing in perchloroethylene vapour at 121°C, then deoxidising by immersion in an alkaline detergent, pickling in HCl, passivation in a chromic acid/H₂SO₄ bath, rinsing in demineralized water and finally drying with N₂ gas. To ensure that the complete chamber was subjected to all stages of all treatments, the four sections were placed in the vacuum brazing oven although it was only the two end sections which were actually brazed to flanges. Once the chamber had been finally welded together it was given a last chemical cleaning in the deoxidising alkaline detergent.

Two 3.6 m long, 131 mm diameter, stainless steel chambers (SS 1 and SS 2) were manufactured from 316 L+N stainless steel tube. They were chemically cleaned first by immersion in perchloroethylene vapour at 121°C, then by immersion in an alkaline detergent at 65°C followed by rinsing in cold demineralized water and finally drying in a vacuum oven at 150°C. As an additional treatment, one chamber, SS 1, was degassed in a vacuum oven at 950°C.

Each test chamber was then vacuum tested by baking at 150°C (Cu and Al) or 300°C (stainless steel) for 24 h. When installed in the synchrotron radiation beam line, all test chambers were baked at 150°C for 24 h after which the base pressure was typically $2 \cdot 10^{-8}$ Pa.

For ease of reference the different treatments are summarized in Table I.

Table I

TEST CHAMBER	TREATMENT
Aluminium	Alkaline etch
Copper	Vapour degreasing
	Alkaline deoxidation
	Acid etch
	Vacuum braze
	Alkaline deoxidation
Stainless steel (SS 1)	Vapour degreasing
	Alkaline detergent
	950°C vacuum degas
Stainless steel (SS 2)	Vapour degreasing
	Alkaline detergent

RESULTS

A. Gas desorption coefficients

The photon induced neutral gas desorption coefficients for Al as a function of the photon dose are shown in Fig. 1. There it can be seen that

Fig. 1. The photon induced neutral gas desorption coefficients for Al as a function of the photon dose.

Fig. 2. The photon induced neutral gas desorption coefficients for Cu as a function of the photon dose.

the gases desorbed are firstly H_2 followed closely by CO and CO_2 , all in the 10^{-2} molecules/photon range, with CH_4 about a factor of 10 lower. With increasing photon dose all desorption coefficients decrease but the CH_4 decreases much faster than the other gases. After a dose of 2×10^{21} photons/m the H_2 has decreased by a factor of 13, CO and CO_2 by a factor of 10 and CH_4 by a factor of 300.

In Fig. 2 the corresponding results for Cu are shown. It can be seen immediately that, compared to Al, the initial desorption coefficients are all about a factor of 10 lower. However the rate of decrease with photon dose is also lower, in that after a dose of 2×10^{21} photons/m the desorption coefficients have only decreased by a factor of 10. Again it is H_2 which has the largest desorption coefficient followed closely by CO and CO_2 with CH_4 lower still.

The results for the stainless steel test chamber (SS 1) which was vacuum degassed at $950^\circ C$ are shown

Fig. 3. The photon induced neutral gas desorption coefficients for stainless steel (SS 1) as a function of the photon dose.

Fig. 4. The photon induced neutral gas desorption coefficients for stainless steel (SS 2) as a function of the photon dose.

desorbed per metre of chamber as a function of photon dose may be obtained. A more practical number is the amount of gas desorbed in Torr litres per metre of chamber, where 1 Torr litre = 3.3×10^{19} molecules. This integration has been carried out for Al, Cu and stainless steel (SS 2) and the results are shown in Fig. 5, 6 and 7 respectively.

in Fig. 3. The initial desorption coefficients are the lowest of all, with H_2 about 1×10^{-3} molecules/photon, CO and CO_2 in the 10^{-4} range and CH_4 in the 10^{-5} range. In common with Cu, the rate of decrease with photon dose is low and both H_2 and CO_2 have decreased by only a factor of about 3.5 and CO and CH_4 by a factor of 10.

Since the 950°C vacuum degassing may be difficult to carry out on large, complex vacuum chambers, this step was omitted from the treatment of test chamber SS 2. The results are shown in Fig. 4 and much to our surprise they were practically identical to those obtained from the vacuum degassed chamber SS 1.

With the advent of non-evaporable getters (NEG) the total quantity of gas desorbed by the synchrotron radiation is important in deciding how much NEG to install and in predicting how often it must be reconditioned during the operation of the machine³. By integration of the desorption coefficients with respect to photon dose, the total number of molecules

Fig. 5. The total quantity of gas desorbed from Al as a function of the photon dose.

Fig. 6. The total quantity of gas desorbed from Cu as a function of the photon dose.

Fig. 7. The total quantity of gas desorbed from stainless steel (SS 2) as a function of the photon dose.

For the Al, which had the highest desorption coefficients, it can be seen that, after 3×10^{21} photons/m, almost 1 Torr l/m of H_2 had been desorbed, 0.1 Torr l/m of CO and CO_2 but only 4×10^{-3} Torr l/m of CH_4 .

After 3×10^{21} photons/m, the Cu test chamber had desorbed 5×10^{-2} Torr l/m of H_2 , 2×10^{-2} Torr l/m of CO_2 , 1.8×10^{-2} Torr l/m of CO and 3×10^{-3} Torr l/m of CH_4 .

For the stainless steel which had the lowest desorption coefficients, after the same photon dose, 3×10^{-2} Torr l/m of H_2 had been desorbed, 1×10^{-2} Torr l/m of CO, 5×10^{-3} Torr l/m of CO_2 and 1×10^{-4} Torr l/m of CH_4 .

Even after 3×10^{21} photons/m, in all the test chambers the quantity of gas desorbed was still increasing and showing only a slight tendency to level off.

Another practical piece of information useful in the design of pumps for vacuum systems exposed to synchrotron radiation is how much gas has to be removed before the desorption coefficients drop below a certain value. These results are shown in Fig. 8, 9 and 10 for Al, Cu and stainless steel respectively.

Fig. 8. The photon induced neutral gas desorption coefficients for Al as a function of the total quantity of gas desorbed.

Fig. 9. The photon induced neutral gas desorption coefficients for Cu as a function of the total quantity of gas desorbed.

Fig. 10. The photon induced neutral gas desorption coefficients for stainless steel as a function of the total quantity of gas desorbed.

B. Photoelectrons

In parallel with the measurements on the photon induced gas desorption, the photoelectron currents in the test chambers were also measured. In the central part of the chamber, a 20 cm long, 1 mm diameter stainless steel wire, running along the axis of the chamber, was suspended from an insulated feedthrough and photoelectrons produced in the vicinity were collected by applying +1000 V to the wire. Applying higher voltages simply collected more photoelectrons from further along the chamber, thus all measurements are relative, but nevertheless give an indication of the photoelectron currents to be expected.

Fig. 11. The measured photoelectron currents in the four test chambers.

The results are shown in Fig. 11 for the four test chambers where it can be seen that the highest photoelectron currents were produced in the Al chambers, followed by the Cu then the stainless steel. With increasing photon dose, as shown by the arrows in the figure, the photoelectron currents decreased. In the stainless steel chambers there was almost no change with photon dose.

C. Extrapolation to LEP

The injection energy of the LEP machine is 20 GeV which corresponds to a critical energy of 5.7 keV for the synchrotron radiation compared to 2.95 keV in DCI. Also, for a large part of the time, LEP would run at 46 GeV where the critical energy is 69.7 keV.

The expression for the desorption coefficient ETA for a given gas is given by:

$$ETA = \frac{3.3 \cdot 10^{19} \cdot \Delta P \cdot S \cdot 2 \cdot \pi \cdot R}{8.1 \cdot 10^{17} \cdot E \cdot I} \quad (\text{Molecules/photon}) \quad (1)$$

where: ΔP is the absolute pressure increase due to the photons (Torr)
 S is the pumping speed for the gas (l/s/m)
 E is the beam energy (GeV)
 I is the beam current (mA)
 R is the bending radius (m).

The vacuum chamber in LEP is made from Al thus we have taken the desorption coefficients from Fig. 1, applied the appropriate LEP pumping speed for each gas, the beam current and the beam energy (either 20 GeV or 46 GeV) and calculated the pressure increase for each of the four gases from equation (1). These absolute pressures were then converted to N_2 equivalent to be able to compare them with the gauges installed in the LEP machine.

No corrections were made for the effect of angle of incidence -11 mrad in DCI and 7 mrad in LEP- nor for the higher critical energy of the photons in LEP. Both these effects would tend to increase the desorption coefficients.

In the LEP machine, H_2 , CO and CO_2 are pumped by NEG strip running practically the whole length of the machine thus the linear pumping speed is well defined. But CH_4 is pumped only by 60 l/s ion pumps placed every 20 m giving a linear pumping speed which changes with distance along the vacuum chamber. The calculations were made with a linear pumping speed for CH_4 of 1 l/s/m, a figure which could easily be in error by more than a factor of two up or down, and, since CH_4 dominates the residual gas due to its low pumping speed compared to the other gases, 1 l/s/m as opposed to about 500 l/s/m, this uncertainty is directly reflected in the total pressure calculation.

The results are shown in Fig. 12 where it can be seen that, despite not having corrected for all the effects mentioned above, the agreement

Fig. 12. A comparison between the predicted and measured pressure increases in the LEP machine.

between the extrapolated and measured total pressures is good and gives confidence that the results from the test chambers may be used to predict the vacuum behaviour in an actual machine with a reasonable degree of accuracy. An idea of the precision in the measured LEP data can be gained from the points just above a dose of 10^{19} photons/m in Fig. 12. A series of four measurements gives a factor of two between the lowest and the highest.

CONCLUSIONS

Under photon bombardment the gases desorbed from all test chambers were, in order of importance, H_2 , closely followed by CO and CO_2 , with CH_4 last. Also, with increasing photon dose, the H_2 , CO and CO_2 desorption coefficients in a given chamber all decreased at about more or less the same rate but the CH_4 decreased faster.

The highest initial desorption coefficients were measured in the Al chamber followed by Cu then stainless steel. The results show that the higher the initial desorption rate the higher is the decrease in the desorption rate. The impression is that, at sufficiently high photon dose, the desorption coefficients for all three metals will tend to similar low values.

Using both 4 keV and 500 eV critical energy photons to measure photon induced gas desorption from Al, Cu and stainless steel, similar results have been found^{4,5}.

The high temperature (950°C) vacuum degassing had little effect on the desorption coefficients despite the fact that such a treatment produces a clean surface as seen by Auger surface analysis and greatly reduces the electron induced gas desorption coefficients⁶.

The photoelectron currents measured in the test chambers showed the same tendency as the desorption coefficients. The highest currents were measured in the Al chamber followed by Cu and then stainless steel. In addition, the photoelectron currents were the same in the two stainless steel chambers. Also in the Al and Cu chambers the photoelectron currents decreased with increasing photon dose. Since electrons contribute to the gas desorption process⁶, the form of the desorption versus photon dose curves reflects both the cleaning of the surface and the reduction in the photoelectron currents.

That the data obtained from test chambers can be used with a reasonable degree of accuracy to predict the vacuum behaviour in a real machine has been verified in the LEP electron positron storage ring. Despite differences in photon critical energy and some uncertainties in pumping speed, the measured and calculated pressures in LEP agreed to within a factor of two. Thus we are confident that our data can be used to better design the vacuum systems of future electron or positron storage rings.

REFERENCES

1. O. Gröbner, A. G. Mathewson, H. Stori, P. Strubin and R. Souchet, *Vacuum* 33 (7), 397, (1983).
2. A. G. Mathewson, *Vuoto*, Vol. XVII, No. 3, 102, (July-September 1987).

3. C. Benvenuti and F. Francia, CERN Report, CERN-LEP-VA/89-61, (1989).
4. S. Ueda, M. Matsumoto, T. Kobari, T. Ikeguchi, M. Kobayashi and Y. Hori, 11th Int. Vac. Congress and 7th Int. Conf. on Solid Surfaces, 25th-29th Sept. (1989).
5. H. J. Halama and C. L. Foester, 11th Int. Vac. Congress and 7th Int. Conf. on Solid Surfaces, 25th-29th Sept. (1989).
6. M-H. Achard, R. Calder and A. G. Mathewson, Vacuum 29(2), 53, (1979).