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A COMPARISON OF THE SYNCHROTRON RADIATION INDUCED GAS DESORPTION
IN ALUMINIUM VACUUM CHAMBERS AFTER CHEMICAL AND ARGON GLOW
DISCHARGE CLEANING

by

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ABSTRACT

In a dedicated synchrotron radiation beam line at the DCI storage ring at Orsay, France a series of measurements were made to evaluate the effect of Argon glow discharge cleaning either as a pre-treatment in the laboratory, or in situ, following the installation and pumpdown of the vacuum system in the LEP (Large Electron Positron Storage Ring) machine. On exposure to synchrotron radiation the gases desorbed were H_2 , CH_4 , CO and CO_2 with Ar in the glow discharge cleaned chambers. With continued exposure the gas desorption decreased with time except that the Ar showed a definite reluctance to clean up. When applied to LEP conditions, chemical cleaning only gave the required beam-gas lifetime after the shortest exposure. The worst overall situation results from the in-situ Ar discharge cleaning.

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INTRODUCTION

When in operation the inner surface of the vacuum system of the electron and positron storage ring LEP¹⁾ is subjected to an intense flux of synchrotron radiation. This radiation, with a power of up to a few kW per metre, is emitted by the circulating relativistic (up to 55 GeV in the first stage of operation) electrons and positrons in the arcs of the machine. The energetic photons and the resulting photoelectrons²⁾ may desorb neutral gas from the surface resulting in large pressure rises detrimental to the functioning of the machine.

In LEP the Al alloy vacuum chambers, after chemical cleaning, are baked in situ to 150°C for 24 hours and, using a combination of sputter ion and non evaporable getter pumps, reach base pressures of 3×10^{-9} Pa.

Unfortunately, there is not a simple relationship between the static pressure (without synchrotron radiation) and the dynamic vacuum (with synchrotron radiation). The static pressure, for a given pumping speed, is determined by the thermal desorption of molecules weakly bound to the surface. The more tightly bound species, which are not removed by the 150°C bakeout and do not thermally desorb at ambient temperature, may however easily be desorbed by the energetic photons (eV to MeV) and/or the associated photoelectrons and can give dynamic pressures which are typically several orders of magnitude above the static base pressure. In addition, the photons which are capable of penetrating into the bulk of the Al wall may also release gas from underlying surface layers. Hence not only the molecules on the surface but also molecules in the surface oxide layer of the aluminium may play a role in the behaviour of the dynamic pressure.

In all cases, all vacuum chambers and components must be subjected to a precleaning to remove gross contamination such as machining oil, grease and particulate matter of all types. A subsequent final cleaning, in the case of aluminium, usually consists of a chemical etch of the surface involving either strong acid or alkaline solutions to remove the contaminated oxide layer.

Such a procedure does not provide an atomically clean surface since traces of the cleaning solutions will always remain and, of course, exposure to air results in adsorption of H₂O, O₂, CO₂ etc. Thus, inevitably, electron storage rings are ultimately cleaned by the synchrotron radiation itself (beam cleaning). This necessitates running the machine for more or less long periods

of time in order to reach a sufficiently low dynamic pressure such that stored beams finally attain a reasonable beam-gas Bremsstrahlung lifetime which, in the case of LEP, is set at 20 hours.

The tightly bound gas molecules may also be removed by subjecting the surface to the energetic ion bombardment of an argon glow discharge, a cleaning method which has been applied successfully to the CERN Intersecting Storage Rings³⁾ and the PETRA e^+e^- storage ring⁴⁾.

This glow discharge treatment can be applied to the vacuum chambers as a pretreatment prior to installation in the machine or as an in situ treatment thus eliminating or much reducing the running time of the machine necessary for self-cleaning.

The purpose of this paper is to evaluate and compare the effect of chemical cleaning alone and, as an addition, Argon glow discharge cleaning either as a pretreatment in the laboratory or in situ following the installation and pump-down of the vacuum chambers in LEP. The evaluation is based on observation of the pressure rise when exposing test chambers to synchrotron radiation in a beam line of the DCI positron storage ring at the LURE laboratory, Orsay, France.

EXPERIMENTAL DETAILS

Vacuum System

The experimental set-up has been described fully in a previous publication¹⁾ but for completeness a brief outline will be given here.

The Al test chambers were pumped via a 72.5 ls^{-1} conductance by a 400 ls^{-1} ion pump and two Ti sublimation pumps. After a 24 hour 300°C bakeout the pressure in the stainless steel pumping system was $\sim 7 \times 10^{-9} \text{ Pa}$ and $\sim 2 \times 10^{-8} \text{ Pa}$ in the test chamber which was baked at only 150°C .

Pressure measurements were made by calibrated Bayard-Alpert gauges and a calibrated quadrupole gas analyser connected to a minicomputer into which was also fed the DCI beam current. With such a system both total and partial pressure changes in both N_2 equivalent or absolute units as a function of the photon flux could be recorded.

A remotely controlled water cooled radiation stopper enabled synchrotron radiation to enter the test chamber or to be turned off at will thus defining the photon dose seen by the test chamber.

Synchrotron radiation

For the measurements reported here the stored positron beam energy was kept at 1.72 GeV which corresponds to a critical energy of 3 keV for the emitted synchrotron radiation spectrum.

The square collimator was dimensioned such that the synchrotron radiation had an angular divergence of 4.7 mrad both horizontally and vertically which attenuated the spectrum below 6 eV.

Only about 15% of photons have energies below 6 eV and are considered ineffective for desorption. Along the length of the test chamber, the beam dimensions varied from 26 mm x 26 mm to 42.5 mm x 42.5 mm.

The maximum current used in an experiment was 180 mA giving a total of about 30 W of power incident on the uncooled test chamber.

Correspondingly, the total flux of photons entering the test chamber is $1 \times 10^{15} \text{ s}^{-1} \text{ mA}^{-1}$.

Each test chamber was 3.5 metres in length and the synchrotron radiation was incident on the side wall of the chamber at a glancing angle of 11 mrad with a spread of ± 2.35 mrad and extended over a length of 3.12 metres.

Test chamber treatment

The chambers were manufactured from an extruded Al tube which has a channel of elliptic cross section for the LEP beam and a rectangular section to house the distributed pump in the form of a non-evaporable getter (NEG) strip as shown in Fig. 1. The chamber material is a precipitation hardenable alloy of the type ISO Al Mg Si (composition Mg 0.35-0.6%, Si 0.3-0.6%, Fe 0.1-0.3%, Cu 0.1%, Mn 0.1%, Cr 0.05%, Zn 0.15%, Ti 0.1%, Al-balance).

All chambers were chemically cleaned according to the following procedure:

1. Vapour degreasing in perchlorethylene vapour at 121°C.

2. Ultrasonic cleaning in an alkaline detergent (pH 9.7, 60°C).
3. Rinsing in cold demineralized water.
4. Immersion in NaOH solution (conc. 40 gl^{-1}) at 45°C.
5. Rinsing by immersion in normal water at 20°C.
6. Immersion in NHO_3 (50% vol.) + 3% vol. HF (40% vol.) at 20°C Rinsing by
7. immersion in demineralized water at 20°C.
8. Drying in hot air at ~ 80°C.

The Argon glow discharge cleaning as a pretreatment after chemical cleaning and before installation in the DCI beam line was performed by inserting a wire along the length of the NEG pumping channel, backfilling the chamber to ~ 1.3 Pa of pure Argon and applying about + 220 V to the wire. The glow discharge penetrated into the beam channel through the interconnecting pumping slots. During the discharge the chamber was heated to 150°C and pumped continuously using a turbomolecular pump with a continuous flow of clean Argon.

The bombarding ion energy was 220 eV and the discharge was continued for sufficient time to give a total argon ion dose of 2×10^{18} ions cm^{-2} . After stopping the discharge, the heating at 150°C and pumping were maintained for 15 hours to degas as far as possible any implanted argon.

The test chamber was installed in the radiation beam line at DCI and rebaked again at 150°C for 24 hours. A final pressure of 2×10^{-8} Pa was obtained.

For convenience, the in-situ Argon glow discharge was also carried out on this same test chamber installed on the DCI beam line after, of course, the first desorption measurements with synchrotron radiation had been made. We assume that any memory effect of the first exposure to synchrotron radiation may be neglected because of the venting to air and the drastic change in surface condition which results from the glow discharge treatment.

The chamber was first exposed to air, repumped and backfilled with pure argon and the discharge was performed as described previously. A limit pressure after this treatment of 1.7×10^{-8} Pa was obtained.

RESULTS

Upon exposure to synchrotron radiation the gases desorbed were H_2 , CH_4 , CO, Ar and CO_2 . Fig. 2 shows typical residual gas spectra before (2a) and while (2b) exposing the test chamber to synchrotron radiation.

In Figs. 3, 4 and 5 are shown the dependence of the photon induced gas desorption in mol. photon⁻¹ as a function of the number of accumulated photons per metre of chamber for the three treatments.

For the chemically cleaned test chamber (Fig. 3) the gases desorbed in order of importance were H_2 , CO_2 , CO and CH_4 .

The cleaning rate of the three leading gases approaches a constant slope whereas the CH_4 cleans up faster.

With Argon glow discharge as a pretreatment (Fig. 4) the gases desorbed in order of importance at the start were again H_2 , CO_2 , CO, CH_4 but in addition also Ar. The CO_2 and CH_4 cleaned up faster than the H_2 and CO. The Ar showed a marked reluctance to clean.

The in-situ glow discharge (Fig. 5) reduced the CO_2 desorption to below 10^{-5} mol. photon⁻¹. In absolute terms all gases show a reduction with respect to the previous results. The initial desorption rates of CO and CH_4 are lower by about factors of 5 and 2 respectively. Again the Argon clean up rate is relatively low as is that of the CO. Both H_2 and CH_4 have slopes very similar to the previous results.

DISCUSSION AND CONCLUSIONS

It would appear at first sight from inspection of Figs. 3, 4 and 5 that the preferred treatment for the LEP Al vacuum chambers would be the in-situ Ar glow discharge since the CO_2 desorption is almost eliminated and the desorption of the other gases significantly reduced. However, the beam-residual gas lifetime in an electron storage ring is determined by Bremsstrahlung on the nuclei of the gas molecules and is not only inversely proportional to the partial pressure of the residual gas but also to the inverse square of the atomic number. In addition the LEP vacuum system, with its non evaporable getter pump, has a high pumping speed for H_2 , CO and CO_2 ⁵⁾ - the active gases - but a very low pumping speed for Ar and CH_4 which are only pumped by 30 ls⁻¹ ion pumps every 20

metres. Thus, to predict the behaviour of the LEP vacuum system from the results of Figs. 3,4 and 5 the differences in pumping speed for the different gases and the photon flux have to be taken into account¹⁾. Also 2.5 mA in DCI at 1.72 GeV produces the same photon flux in the test chamber at almost the same critical energy as 1 mA in LEP at 20 GeV¹⁾.

Taking into account these factors, the product of the beam residual gas lifetime in hours and machine current in mA has been calculated⁶⁾ for LEP and is shown in Fig. 6 for the three chamber treatments. For convenience, the photon dose has been expressed in terms of mA hours of LEP operation (beam dose).

At low beam dose there is little difference between the chemical cleaning alone and the Ar glow discharge as pretreatment. However, with increasing beam dose the discharge treatment falls behind due to the continual desorption and relatively slow clean up rate of the high atomic number Ar implanted in the surface during the glow discharge pretreatment.

The effect of this implanted Ar is even more evident when the discharge is applied in situ. The lifetime after this treatment is about a factor of two higher at the start when compared to the other treatments but increases only very slowly.

In conclusion, therefore, for the LEP machine the best among the 3 treatments is the chemical cleaning alone followed by the Ar glow discharge as pretreatment and the in-situ Argon glow discharge the worst due to the low Ar pumping speed.

It is, of course, not excluded that discharge treatments with other low atomic number gases such as He⁶⁾ or N₂⁷⁾ could give better results.

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REFERENCES

1. O. Gröbner, A.G. Mathewson, H. Störi and P. Strubin, Vacuum 33, 397 (1983).
2. E. Alge, O. Gröbner, M. Kobayashi, A. Mathewson, R. Souchet and P. Strubin, CERN LEP Vacuum Group Technical Note, LEP-VA/AM/sm, 10th February 1984, CERN 1211 Geneva 23, Switzerland.
3. R. Calder, A. Grillot, F. Le Normand and A.G. Mathewson, CERN/ISR-VA/77-59, 1977, CERN, 1211 Geneva 23, Switzerland.
4. A.G. Mathewson, J. Kouptsidis and L. Hipp, Proc. 7th Int. Vacuum Congress and 3rd Int. Conf. on Solid Surfaces, Vienna, p. 235, 1977.
5. C. Benvenuti, Nucl. Instr. and Meth. 205, 391, (1983).
6. H. Störi, Vacuum 33, 171, (1983).
7. N. Hilleret, IX International Conference of Solid Surfaces, Madrid, 26 Sept. - 1 Oct. 1983.

Figure 1 : Al test chamber showing the pumping slots, discharge wire and the synchrotron radiation

Fig. 2a : The residual gas spectrum before exposing the test chamber to synchrotron radiation

Fig. 2b : The residual gas spectrum during exposure to synchrotron radiation

Figure 3 : The synchrotron radiation reduced gas desorption at 11 mrad in mol. photon⁻¹ as a function of the accumulated number of photons incident on one meter of chamber after chemical cleaning.

Figure 4 : The synchrotron radiation induced gas desorption at 11 mrad in mol. photon⁻¹ as a function of the accumulated number of photons incident on one meter of chamber after the glow discharge pretreatment.

Figure 5 : The synchrotron radiation induced gas desorption at 11 mrad in mol. photon⁻¹ as a function of the accumulated number of photons incident on one meter of chamber after the in situ glow discharge treatment.

Figure 6 : The product of the beam-residual gas lifetime in hours and the LEP beam current in mA at 20 GeV beam energy as a function of the LEP beam dose in mA hours.