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Synchrotron Radiation-Induced Desorption from a NEG-Coated Vacuum Chamber

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Abstract

When the whole inner surface of a vacuum chamber is coated with a non-evaporable getter film, very low static and dynamic pressures are expected after activation. In an accelerator environment this could result in a longer beam lifetime, in a lower risk of pressure bumps, and in a lower level of bremsstrahlung radiation due to the beam-gas interactions. To substantiate these favourable characteristics a Ti-Zr-V coated stainless steel chamber has been tested on a dedicated beamline at the ESRF. It is shown that a large reduction of the synchrotron radiation-induced desorption occurs after activation.

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1.   INTRODUCTION

Vacuum systems of particle accelerators have to be designed so as to maintain a pressure in the beam pipe low enough to ensure that the beam lifetime is always longer than that defined in the design and induced radioactivity is always lower than that allowed by the personal safety standards.

Recently a new pumping solution for the vacuum system of particle accelerators has been developed and studied at CERN [1,2]. It consists in coating the vacuum chamber ex-situ with a μm thick non-evaporable getter (NEG) film. The activation of the film is then performed during the in-situ bake-out of the vacuum system. Many elements and alloys have been tested (essentially those of the 3rd and 4th group of the periodic table) but the Ti-Zr-V combination is by far the one with the lowest activation temperature (about 180 °C for a 24 hours long bake-out) [3].

The potentialities of this solution in reducing synchrotron induced desorption seemed clear from the beginning.

NEG coatings offer two advantages compared to the standard approach. First of all the whole inner surface of the chamber can be coated therefore allowing a large increase of the pumping speed per meter of chamber after activation. On the other hand during activation the surface oxide and contaminants are dissolved into the bulk therefore giving rise to a very clean surface which should have a lower photon desorption yield as has already been proved for electron stimulated desorption [4].

To substantiate these potential benefits a Ti-Zr-V coated chamber has been tested in a devoted synchrotron light line at the European Synchrotron Radiation Facility (ESRF).

2.   EXPERIMENTAL SET-UP

The chamber fabricated for the experiment was a 316LN, 60 mm diameter, 2 m long tube provided with a double wall for water-cooling. The chamber had been cleaned by applying the CERN standard procedure [5] and then vacuum-fired at 950 °C for 2 hours.

The chamber was coated using the cylindrical magnetron configuration. The axial cathode was made by twisting together three 1-mm diameter wires of Ti, Zr and V (about 99.8 % purity). Pure Ar at a pressure of about 10^{-2} mbar has been used to generate the plasma. The cathode potential was –500 V during the deposition with the chamber grounded. The magnetic field, which was generated by an external solenoid, had an intensity of about 150 Gauss. The sputtering rate was of the order of 1 Å s^{-1}, and the final thickness of the coating was about 2 μm.

Before its installation in the ESRF line, the chamber had been stored under dry nitrogen for some days after venting.

At the ESRF, the D31 beamline has been used for the experiment [6]. The main parameters of the ESRF storage ring and of the synchrotron light provided to the D31 beamline are summarised in table 1. A schematic view of the line is shown in Figure 1.

An 8 mm high, 60 mm wide stainless steel diaphragm was interposed between the experimental chamber and the rest of the beamline vacuum system. The diaphragm, which was coated on the side facing the chamber, was designed to prevent large photon backscattering from the experimental chamber. A water-cooled flange, which was also coated, closes the chamber at the opposite side.

Pressures in the system were measured by Penning and Bayard-Alpert gauges on both sides of a 19.1 l/s (N_2-equivalent) conductance aperture (4 mm high, 54 large, on a 2 mm thick copper disc).

Pumping was provided by a 400 l s^{-1} ion pump and by a 2000 l s^{-1} Ti sublimation pump located between the line insulation gate valve and the aperture.

An indication of the residual gas composition can be obtained by means of a quadrupole gas analyser (not calibrated) mounted between the conductance and the chamber. A shutter and a gate valve were installed between the experimental line and the front-end on one side and the storage ring on the other.

The incident angle of the synchrotron light was usually set at approximately 25 mrad, but it could be changed remotely during the experiment by means of a stepped motor from 0 to 50 mrad. Before the measurement all the line components had been baked at about 300 °C. During bake-out the coated chamber was heated only at 100 °C to desorb water vapour without activating the chamber therefore preventing pumping of gas released by the rest of the system.

After cooling at room temperature the total pressures were of the order of 10^{-11} mbar on the pumps' side of the system and of the order of 10^{-10} mbar on the chamber's side. As usual, H_2 was the leading gas.

The activation of the coating was performed by heating the chamber at 250 °C for about 20 hours while keeping the rest of the system at room temperature. After activation, the pressure recorded on the chamber's side dropped by a factor of 10, becoming lower than the pressure recorded on the pumps' side, where the effect of activation is only small.

During bombardment the chamber and the end flange were water-cooled. The same chamber and end flange could also be cooled with liquid nitrogen transported from a dewar, placed outside the main ring, by mean of a dedicated cryogenic line.

3.   EXPERIMENTAL RESULTS

The desorption yields of the unactivated coating were measured by bombarding the chamber for a short time (about one hour) in order to prevent unduly radiation cleaning of the wall of the chamber. The total desorption yield at the very beginning is about 10^{-2} molecules per photon. Measurements performed in the past on bare stainless steel and copper gave values [7] of the same order.

The higher dose data can be fitted, as usual, by a power function of the dose. The exponential coefficient of the power law is equal to –1, which is in the upper range of the coefficients normally obtained for standard materials (in general between -0.6 and -1)[7]. The largest signal intensity corresponds to that of mass 2, followed by mass 28 (3 times lower), 15, 16 and 44 (each one 10 times lower).

When the chamber is rotated to the straight position so that only the end flange is bombarded by photons, no changes in the total desorption flux and in the mass spectrometer peaks were recorded.

After activation a sharp decrease (about a factor 380) of the total desorption occurred as shown in Figure 2 and, as a consequence, a value of the desorption yield of about 7 x 10^{-6} molecules per photon is obtained after only some minutes of bombardment. Such a low value can also be obtained with standard materials but only after months of bombardment [7].

The total desorption yield after activation can again be well fitted by a power function of the dose but with a lower exponential factor, namely -0.38. The bombardment is continued up to a total dose of 1.2 x 10^5 mA h. At this final value of dose, the total desorption yield is in the high 10^{-7} molecules per photon range.

The intensities of the quadrupole gas analyser peaks underwent a similar decrease after activation, but the ratios of the intensities before and after activation were unequal for the different peaks. CO and CO_2 peak intensities decreased by a factor 400, the mass 2 peak signal corresponding to H_2 was reduced 300 times while CH_4, C_2H_6 and C_3H_8 typical peaks suffered a decrease by a factor 200.

Bombarding the chamber in the straight position at a dose of 5×10^3 mA h resulted in a 33 % decrease of the total desorbed flux. H_2, CO and CO_2 peaks experienced a 36 %, 15 % and a 20 % reduction of their intensities. On the other hand the intensities of the CH_4 peaks increased by a factor 3.6.

At the end of the measurement the chamber was cooled down to liquid nitrogen during bombardment. The cooling was effective in reducing the total degassing by a factor two. The CO peak intensity remained nearly constant while that of H_2 was reduced by a factor two. The ratio between the H_2 and the CH_4 peaks, which during all the experiment were lower than 10, became as large as 40 during the liquid nitrogen cooling.

4.   DISCUSSION

The decrease of the measured desorption yield of a NEG surface after activation is always the result of two different causes. On the one hand the intrinsic desorption yield of the surface decreases because the surface oxide layer is dissolved into the bulk during activation leaving a much cleaner surface. On the other hand molecules leaving the NEG surface have a probability to be repumped by the surface that depends not only on the sticking probability of the NEG surface but also on the geometry of the system.

A Monte-Carlo simulation of the D31 set-up allows the two contributions to be disentangled if the sticking probabilities of the NEG surface can be measured independently. This independent measurement can be done only if a constant source of gas is available in the chamber. This unfortunately can be hardly carried out without perturbing the chamber environment and so the total gas-load during bombardment. Therefore sticking probability values obtained with a dedicated laboratory system were assumed in the simulation.

It has been measured [3] that for a fully activated Ti-Zr-V surface the sticking probabilities are in the of 10^{-1} for CO and 10^{-3} for H_2 range and that even for a partially activated surface the CO sticking probability is always about a factor 100 larger than that of H_2.

As a consequence the Monte-Carlo simulation shows that CO degassing should drop by a factor larger than 2000 after activation while a reduction by a factor ranging between 5 and 50 should be expected for H_2 by the effect of the wall pumping alone (see Figure 3). The global reductions have to be much larger than those calculated above due to the reduction of the intrinsic desorption.

Because the measured reduction for CO is only 400 we are forced to assume that after the activation most of the CO molecules are generated outside the coated chamber and that those coming from the chamber contribute such a low amount to the gas load that they can be neglected.

It can be shown that, if the desorption yields of the inactive Ti-Zr-V surface and of the stainless steel vacuum system walls are similar, about one hundredth of the total number of photons hitting outside the chamber gives rise to the measured reduction factors for CO.

The same assumption can also be applied to H_2 simply by taking into account that the capture probability of the chamber diaphragm aperture is about 0.5 for this gas instead of about 1 for CO.

Because CH_4 cannot be pumped by NEG's, its reduction factor can be ascribed only to the reduction of the intrinsic desorption. The experimental result indicates that this is at least a factor 200.

The presence of an external source of gas is substantiated by the results obtained during the straight direction bombardment and the liquid nitrogen cooling. Monte-Carlo simulation shows that molecules generated at the end flange have a much lower probability to escape from the chamber than those generated uniformly along the tube wall. For instance for a sticking probability of 10^{-2} the probability to escape is more than 20 times higher in the latter case. Therefore it can be settled that by turning the chamber to the straight position while bombarding a huge decrease of the RGA signals should be recorded except for those corresponding to CH_4 which is not pumped. In spite of that the experimental results show only a partial decrease of the signal. On the contrary the CH_4 peaks increased probably because the accumulated dose of the end flange is lower (unfortunately by an unknown amount) than that received by the chamber.

When the chamber is cooled with liquid nitrogen a decrease by about a factor 10 is expected for the H_2 peak if this gas comes entirely from the chamber because, as it is well known, its sticking probability increases by about a factor 10 on the cold getter surface [8].

But the results show that a reduction of only a factor two is obtained, which could be explained by the increase of the capture probability of the diaphragm from 0.5 to 1 for the gas generated outside the chamber.

Other less direct evidence for the dominance of an external source after activation is given in Figure 2 where if the dose scale of the activated-case portion of the plot is compressed by about a factor 100, the slopes of the curve before and after activation are quite similar.

5. CONCLUSIONS

A reduction of the total degassing produced by synchrotron radiation bombardment by at least a factor 380 has been measured for a Ti-Zr-V coated stainless steel chamber after activation. This has been obtained in a very short time compared to that needed for the conditioning of standard materials.

Some evidences exist that a large part of the remaining desorption after activation could be due to a small fraction of the photon flux striking outside the chamber. New measurements are needed to consolidate this conclusions.

An aluminium coated chamber will soon be tested to verify if the Ti-Zr-V film performances are affected by a different substrate material. In parallel 5 m long chambers will be coated and mounted in the ESRF main ring to test them in a real accelerator environment and to collect other data that cannot be obtained in a beam line, like for example the effect of the coating resistance on the electron beam wall impedance.

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Table 1 -   Fundamental parameters of the ESRF main ring and of the D31 beamline

Synch. Radiation Critical Energy	20.5 keV
Angular Acceptance	4.234 mrad
Photon Flux (E>10 eV)	2.94×10^{15} photons s^{-1} mA^{-1}
e$^-$ Beam Energy	6 GeV
Typical e$^-$ Beam Current	185 mA

Figure 1 - Schematic view of the D31 beamline vacuum system

Front-End Slits
and Absorber

PG

SIP

Gate Valve

BAG PG

SIP

SP

Turbomolecular
pumping group

QMA

Conductance Diaphragm

PG BAG

Water or liquid nitrogen
cooling circuit

NEG-coated chamber

PG : Penning Gauge
BAG : Bayard-Alpert Gauge
QMA : Quadrupole Masss Analyser
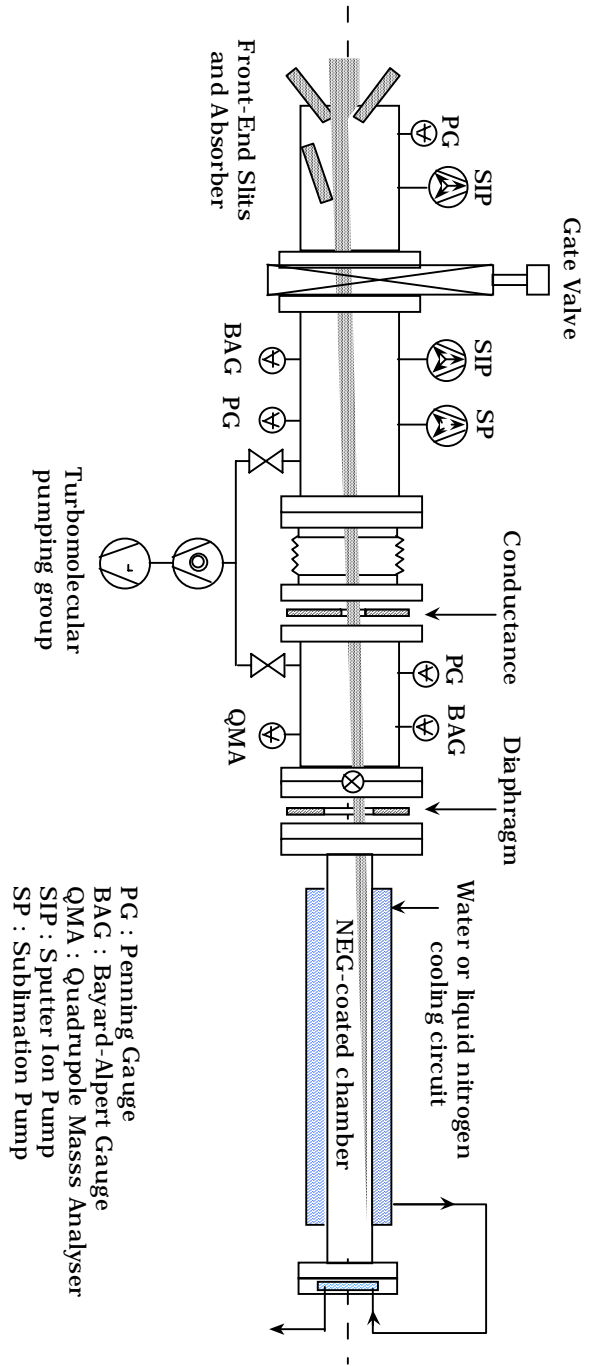SIP : Sputter Ion Pump
SP : Sublimation Pump

Figure 2 - Total molecular desorption yield η (N_2 equivalent) of the Ti-Zr-V coated stainless steel chamber as a function of the accumulated dose before and after activation
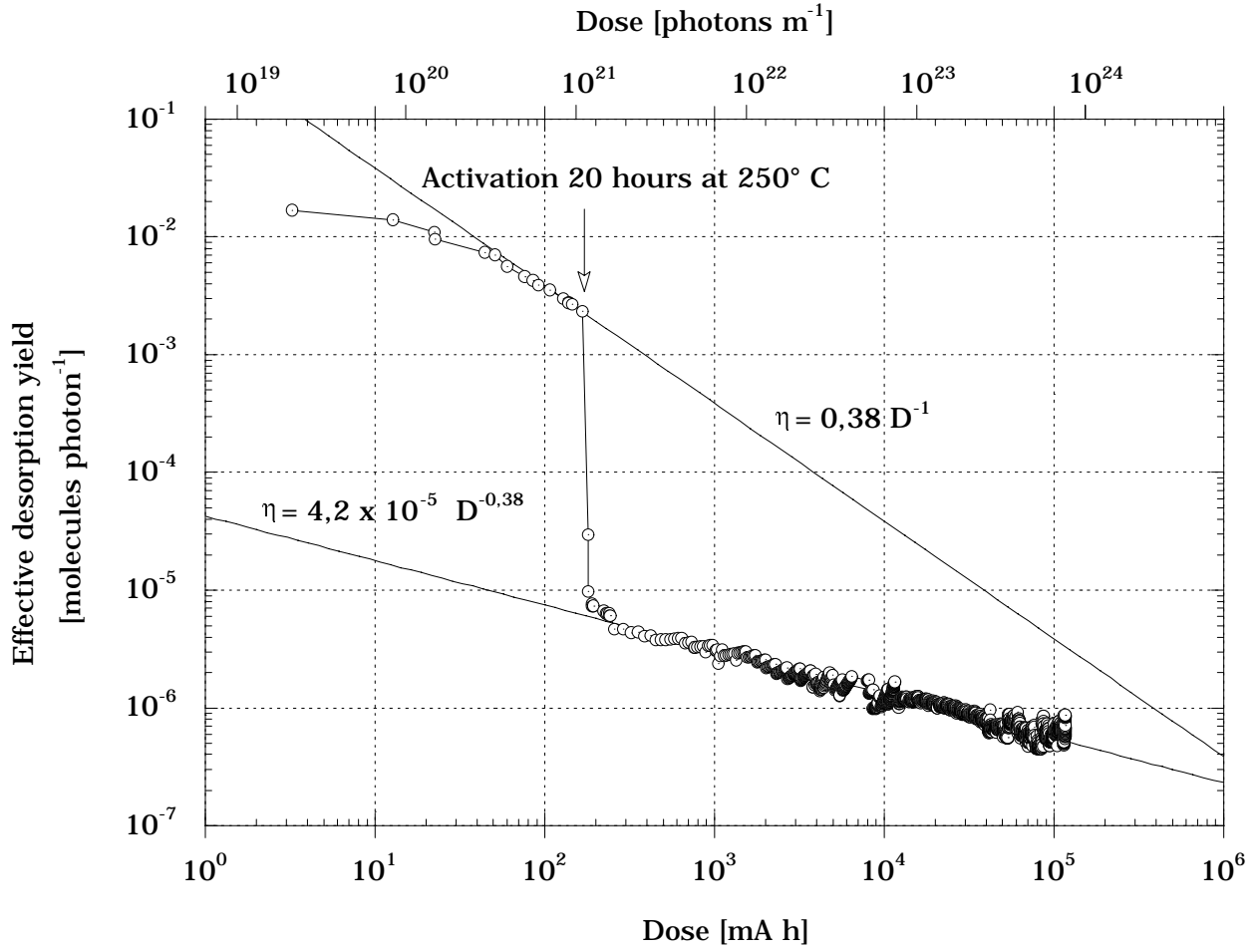
Figure 3 - Pressure reduction factor as a function of the coating sticking probability obtained by Monte-Carlo simulation assuming a constant gas load before and after activation.