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Ion-stimulated gas desorption yields of electropolished, chemically etched, and coated (Au, Ag, Pd, TiZrV) stainless steel vacuum chambers and St707 getter strips irradiated with 4.2 MeV/u lead ions

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The ion-induced desorption experiment, installed in the CERN Heavy-Ion Accelerator LINAC 3, has been used to measure molecular desorption yields for 4.2 MeV/u lead ions impacting under grazing incidence on different accelerator-type vacuum chambers. Desorption yields for H_2, CH_4, CO, and CO_2, which are of fundamental interest for future accelerator applications, are reported for different stainless steel surface treatments. In order to study the effect of the surface oxide layer on the gas desorption, gold-, silver-, palladium-, and getter-coated 316 LN stainless steel chambers and similarly prepared samples were tested for desorption at LINAC 3 and analyzed for chemical composition by x-ray photoemission spectroscopy. The large effective desorption yield of 2×10^4 molecules /Pb^{53+} ion, previously measured for uncoated, vacuum fired stainless steel, was reduced after noble-metal coating by up to 2 orders of magnitude. In addition, pressure rise measurements, the effectiveness of beam scrubbing with lead ions, and the consequence of a subsequent venting on the desorption yields of a beam-scrubbed vacuum chamber are described. Practical consequences for the vacuum system of the future Low Energy Ion Ring are discussed.

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I. INTRODUCTION

A dynamic pressure rise induced by the loss of heavy ions has been seen first in 1997 during Pb^{54+} accumulation and cooling tests in the Low Energy Antiproton Ring (LEAR) at CERN [1]. Beam-induced desorption limited the beam lifetime and the intensity in LEAR. The vacuum degradation observed was not understood at that time. In preparation of the heavy-ion physics with the Large Hadron Collider (LHC) at CERN, LEAR has to be converted into a Low Energy Ion Ring (LEIR). An average dynamic pressure of 3×10^{-12} Torr is required in LEIR to satisfy the requested 30 s beam lifetime [2]. An experimental program, aiming to prepare the LEIR vacuum system and to understand the desorption phenomena, was started at CERN in November 2000 [3]. Molecular desorption yields of up to 2×10^4 molecules/Pb^{53+} ion were measured for stainless steel surfaces, after ~100 h of beam scrubbing the yield was reduced by 2–3 orders of magnitude [4]. During 2001 and 2002 we tried to explore a pragmatic solution to reduce the desorption yields without relying on the beam scrubbing alone. The experiments described in this paper focused mainly on the question of whether the damaged surface layer and/or the native oxide layer of 316 LN stainless steel influence the molecular desorption of heavy ions with an energy of 4.2 MeV/u. In parallel to our studies, large pressure rises and high desorption yields were also reported from GSI [5] and BNL [6,7].

II. EXPERIMENTAL SETUP

Heavy-ion-induced molecular gas desorption of coated stainless steel vacuum chambers was measured in LINAC 3. The beam line to steer the lead ion beam into the experimental setup and the experiment itself have been fully described in an earlier article [4]. A picture of the experimental area is shown in Fig. 1.

Five 1.4 m long, $\varnothing_i = 145$ mm, test vacuum chambers (I, E, F, K, L) were manufactured from 316 LN stainless

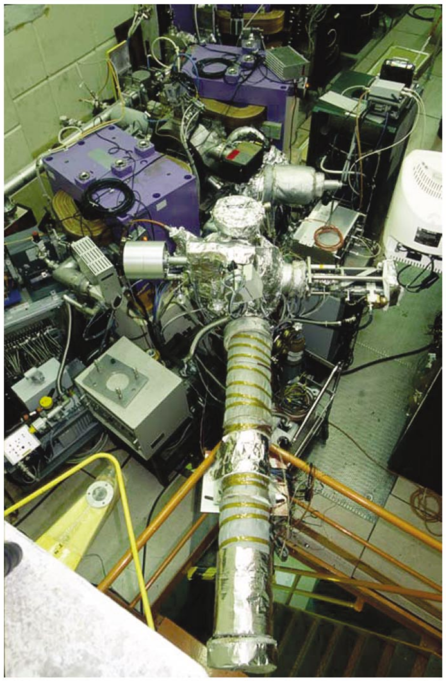

FIG. 1. (Color) Picture of the CERN ion-induced desorption experiment in LINAC 3. The 1.4 m long test vacuum chamber is visible in the foreground.

steel and chemically cleaned, first by degreasing at 50 °C with ultrasonic agitation, followed by rinsing in cold demineralized water, rinsing with alcohol, and finally drying with hot air at 80 °C. Apart from the standard cleaning procedure described above the following surface preparations were performed:

Chamber I: Test #1: chemical etching (50 μm), vacuum firing (1050 °C, 2 h), *in situ* baking (300 °C, 24 h).
Chamber I: Test #2: venting with neon gas, end flange dismantled (1 h), *in situ* baking (300 °C, 24 h).
Chamber I: Test #3: Ni coating (2 μm), Ag coating (2 μm), *in situ* baking (300 °C, 24 h).
Chamber E: Test #1: electropolishing (50 μm), vacuum firing (950 °C, 2 h), *in situ* baking (300 °C, 24 h).
Chamber E: Test #2: electropolishing (150 μm), vacuum firing (950 °C, 2 h), *in situ* baking (400 °C, 24 h).
Chamber E: Test #3: Ni coating (2 μm), Au coating (30 μm), *in situ* baking (300 °C, 24 h).
Chamber F: chemical etching (50 μm), vacuum firing (950 °C, 2 h), *in situ* baking (300 °C, 24 h).
Chamber K: vacuum firing (950 °C, 2 h) of the chamber and a perforated 316 LN stainless steel liner, spot welding of ~25 m St707 getter strips (30 mm width) inside the liner, liner insertion into the vacuum chamber, *in situ* baking (350 °C, 24 h + 400 °C, 3 h).
Chamber L: electropolishing (150 μm), vacuum firing (950 °C, 2 h), Pd sputtering (0.6 μm), *in situ* baking (300 °C, 24 h).

For all test chambers, the assembled end flanges were prepared exactly in the same way as the corresponding vacuum chamber. A picture of vacuum chamber K with the inserted St707 getter strips, which were spot welded onto a stainless steel liner, is shown in Fig. 2.

FIG. 2. (Color) Picture of vacuum chamber K, equipped with a perforated 316 LN stainless steel liner. Inside the liner about 25 m St707 getter strips (30 mm width) are spot welded.

III. RESULTS

A. Single shot desorption yields

Effective ion-induced desorption yields $\eta_{\mathrm{eff},ss}$ have been measured for a fixed impact angle of $\theta = 89.2°$ with a single shot (560 μs long) technique [4] using 4.2 MeV/u Pb^{53+} ions. The effective desorption yield is given by

$$\eta_{\mathrm{eff},ss} = G \times \frac{\Delta P \times V}{N_{\mathrm{Pb}}},$$

where ΔP is the partial pressure increase after one shot, V is the test volume, N_{Pb} is the number of impacting lead ions, and G is a constant. The results obtained in this study are summarized in Fig. 3 and Table I.

The single shot measurements of the bare stainless steel surfaces (I#1, E#2) are all described by a fast partial pressure increase after the impact of the Pb ions, followed by a slow decay determined by the pumping speed of the vacuum system. The coated stainless steel chambers (I#3, E#3, L) and the St707 getter (K) show a different single shot behavior. After the lead ion impact the pressure inhibits a sharp maximum. This peak value was found to be always larger for the coatings than for the bare stainless steel surfaces. Nevertheless, the partial pressure decrease of the coated chambers is much faster than for the uncoated chambers indicating a pumping of the coatings (Au, Ag, Pd) and the St707 getter. As already observed in our previous study [4], the main molecules desorbed were CO followed by $\mathrm{CO_2}$, $\mathrm{H_2}$, and $\mathrm{CH_4}$. CO and $\mathrm{CO_2}$ together represented about 86%–91% of the desorbed species. It is surprising that the Ag and Pd coatings show a higher single shot desorption yield than the uncoated surfaces.

B. Beam scrubbing measurements

All five vacuum chambers have been continuously (every 1.2 s) bombarded with Pb^{53+} ions under 89.2° grazing incidence. In total, nine different scrubbing tests were performed. With the measured pressure increase ΔP the effective desorption yield η_{eff} (molecules/ion) is then given by

$$\eta_{\mathrm{eff}} = \frac{\Delta P \times S}{\dot{N}_{\mathrm{Pb}} \times k_B \times T} = G \times \frac{\Delta P \times S}{\dot{N}_{\mathrm{Pb}}},$$

where S is the pumping speed in ℓ/s and $\dot{N}_{\mathrm{Pb}}$ is the number of impacting lead ions per second. The beam scrubbing results for vacuum chamber I are summarized in Fig. 4.

For the uncoated chamber (I#1) an effective desorption yield of ~7300 molecules/ion was measured at the start of the scrubbing run, this value decreased by a factor of 38 after the impact of about 1.3×10^{12} ions/cm^2 during 80 h. For the second test (I#2), we vented the vacuum chamber after the first beam cleaning, dismantled (under continuous Ne gas flow) the end flange for 1 h, reinstalled the flange, pumped and baked again at 300 °C for 24 h. As can be seen in Fig. 4, the beam scrubbing effect was found to be completely lost and it needed again the same ion dose as in the first test to beam clean the vacuum chamber.

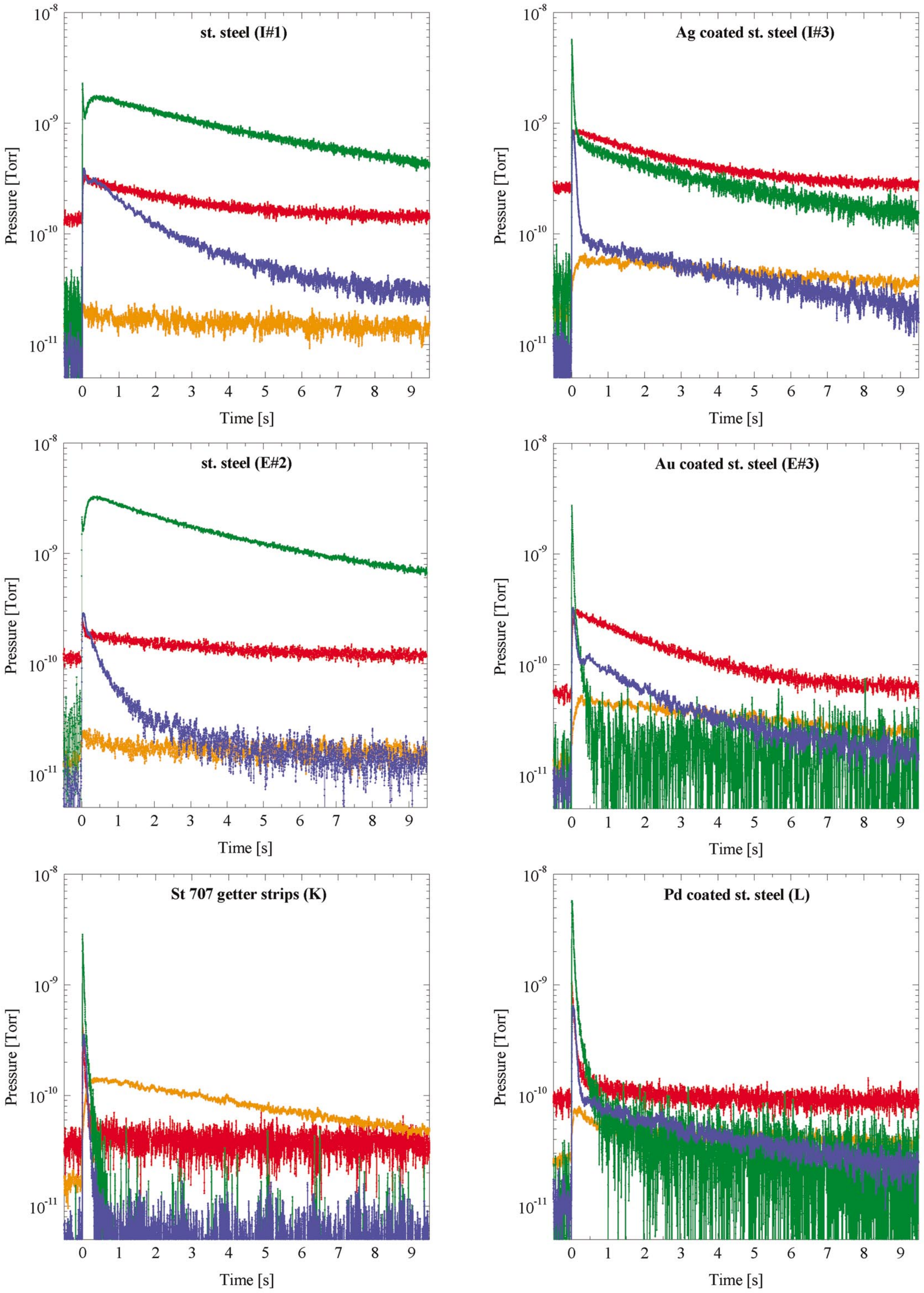

FIG. 3. (Color) Single shot partial pressure rises of H_2 (red), CH_4 (orange), CO (green), and CO_2 (blue) measured for six different vacuum chambers using pulses of 1.5×10^9 Pb^{53+} ions impacting under 89.2° grazing incidence.

Finally, chamber I#2 was coated with a 2 μm thin silver film (I#3), baked (300 °C, 24 h), and measured again (see Fig. 4). The effective desorption yield decreased to ~1500 molecules/ion, which corresponds to an improvement factor of 4.9 due to the Ag coating.

The obtained scrubbing results for vacuum chamber E are also shown in Fig. 4. The scrubbing behavior for the 50 μm electropolished (E#1) and the 150 μm electropolished chamber (E#2) was found to be identical, although the latter one was *in situ* baked at 400 °C instead

TABLE I. Effective desorption yields $\eta_{\mathrm{eff},ss}$ of the main desorbed molecules measured for 4.2 MeV/u Pb^{53+} ions impacting under $\theta = 89.2°$ grazing incidence. Normal incidence corresponds to $\theta = 0°$.

		Effective desorption yields $\eta_{\mathrm{eff},ss}$ [molecules/ion]				
Chamber	Coating	H_2	CH_4	CO	CO_2	Total
I #1	⋯	274	206	2914	469	3863
I #2	⋯	⋯	⋯	⋯	⋯	⋯
I #3	Ag (2 μm)	850	55	7231	1087	9223
E #1	⋯	146	21	4344	552	5063
E #2	⋯	107	15	4040	383	4545
E #3	Au (30 μm)	370	53	4008	403	4834
F	⋯	140	24	3853	386	4403
K	⋯	581	186	4145	488	5400
L	Pd (0.6 μm)	1323	71	7603	844	9841

of 300 °C. For E#2, the effective desorption yield of ~11 000 molecules/ion, measured at the start of the scrubbing run, was about 50% higher than for the 50 μm chemically etched chamber I#1. After the first two sets of measurements chamber E#2 was coated with a 30 μm thick gold layer (E#3) and measured again (see Fig. 4). As already observed for the Ag coated chamber, also the Au coating reduced the desorption yield by a factor of 6.9 to ~1600 molecules/ion. The time needed to reach the maximum of ΔP, which is equivalent to the maximum of

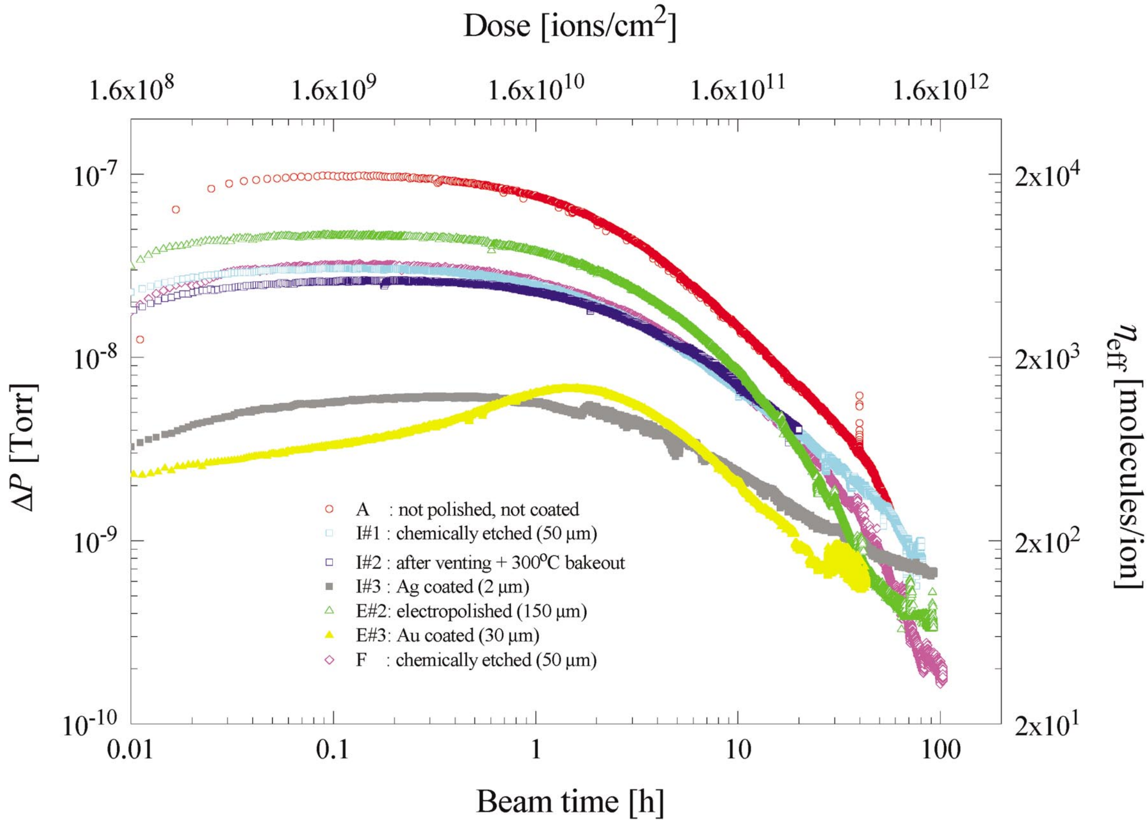

FIG. 4. (Color) Beam cleaning measurements for four different 316 LN stainless steel vacuum chambers continuously bombarded with ~1.5×10^9 Pb^{53+} ions (per shot) under $\theta = 89.2°$ grazing incidence. The results obtained for a chemically etched vacuum chamber (I#1), which was vented after the first scrubbing run, measured again (I#2), and finally 2 μm Ag coated (I#3), are compared with a second chemically etched chamber (F), an electropolished (E#2) and 30 μm Au coated vacuum chamber (E#3). For comparison, the scrubbing of the previously studied LEAR-type (not polished, not coated) vacuum chamber (A) is also displayed [4].

η_{eff}, was found to be larger for the Au coated chamber than for all other vacuum chambers studied so far at LINAC 3.

We currently have no sound explanation for this experimental observation. The measured pressure rise and beam scrubbing of chamber F, which was etched and vacuum fired at 950°, is displayed in Fig. 4. An effective desorption yield of about 7600 molecules/ion was measured at the beginning of the scrubbing run, a yield very close to the value obtained with chamber I#1, which was also etched but vacuum fired at 1050°. After 100 h heavy-ion bombardment of chamber F the molecular desorption decreased by a factor of ~170. In conclusion, there is no benefit to vacuum fire accelerator-type stainless steel vacuum chambers at 1050 °C, instead of the normally used 950 °C, in order to decrease the effective ion-induced desorption yield.

In Fig. 5 the scrubbing results for the Pd sputter coated vacuum chamber L and the St707 getter strips are compared with the Ag and the Au coated chambers. For comparison, the previously obtained results for a nonevaporable getter (NEG) coated (TiZrV) vacuum chamber, that was activated first at 200 °C and later at 300 °C [4], are also shown in Fig. 5 together with the scrubbing of the LEAR-type vacuum chamber A [4]. The measured pressure increase for the Pd coating and the getter strips were about 2 orders of magnitude lower than for the unpolished/uncoated stainless steel vacuum chamber. The reduced dynamic pressure rise of the Pd film was found to be identical with that of the NEG coating which was activated at 200 °C for 24 h (see Fig. 5). The heavy-ion-induced molecular desorption of the St707 getter was found to be very similar to the sputter coated Pd film. During beam scrubbing of the Pd surface a pressure fluctuation was observed after 15 h, which became less pronounced after 40 h of continuous ion bombardment. This pressure fluctuation, also observed for the getter strips, the Au coated and some other vacuum chambers, remains unexplained for the moment.

At the beginning of each scrubbing run the total pressure increase ΔP, due to continuous (every 1.2 s) bombardment with Pb^{53+} ions under 89.2° grazing incidence, was measured. In Fig. 6 the results obtained during this study (5 chambers, 9 surfaces) and a previously published paper (5 chambers, 6 surfaces) [4] are summarized. It is demonstrated that the large pressure rises of either bare or glow discharged 316 LN stainless steel vacuum chambers was reduced by means of electropolishing or chemical etching,

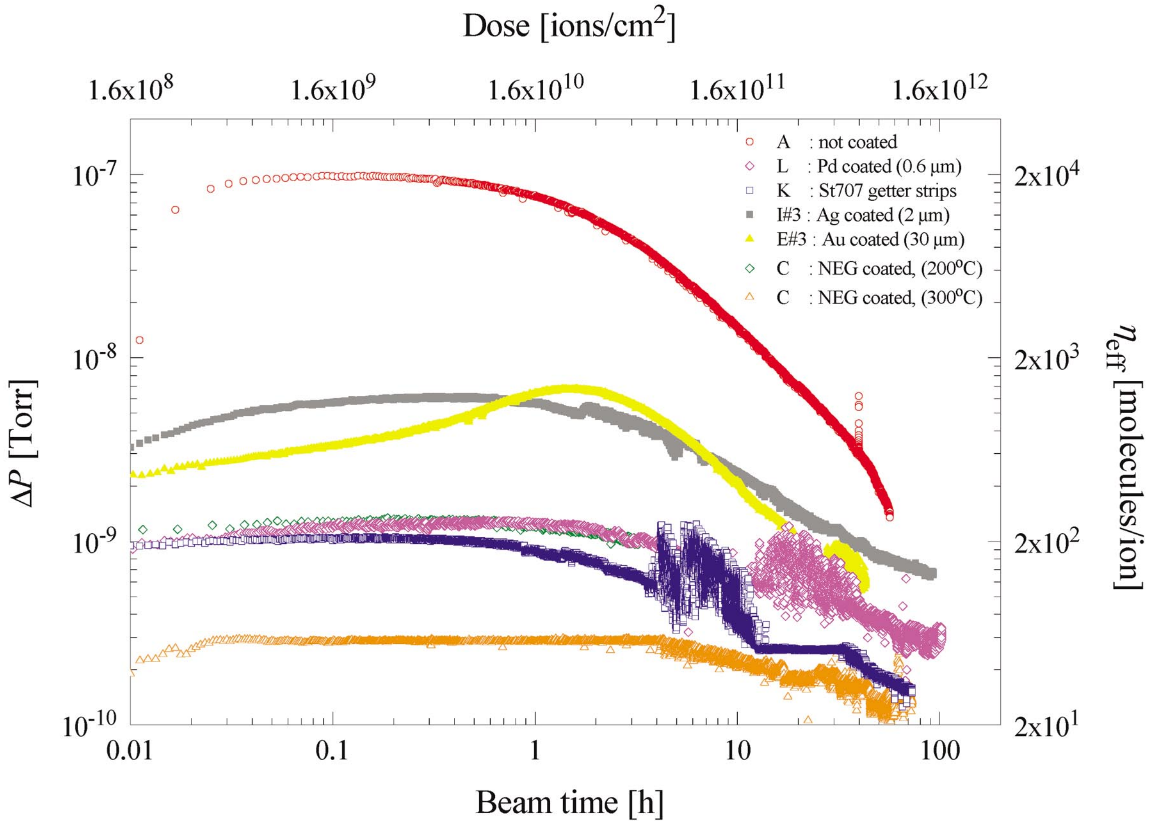

FIG. 5. (Color) Beam cleaning measurements for six different 316 LN stainless steel vacuum chambers continuously bombarded with $\sim 1.5 \times 10^9$ Pb^{53+} ions (per shot) under $\theta = 89.2°$ grazing incidence. The result for the Pd coating (L) and the St707 getter strips (K) are compared with the Ag (I#3) and Au (E#3) coatings. For comparison, the scrubbing of the previously studied NEG coating (C) and the LEAR-type vacuum chamber (A) are also shown [4].

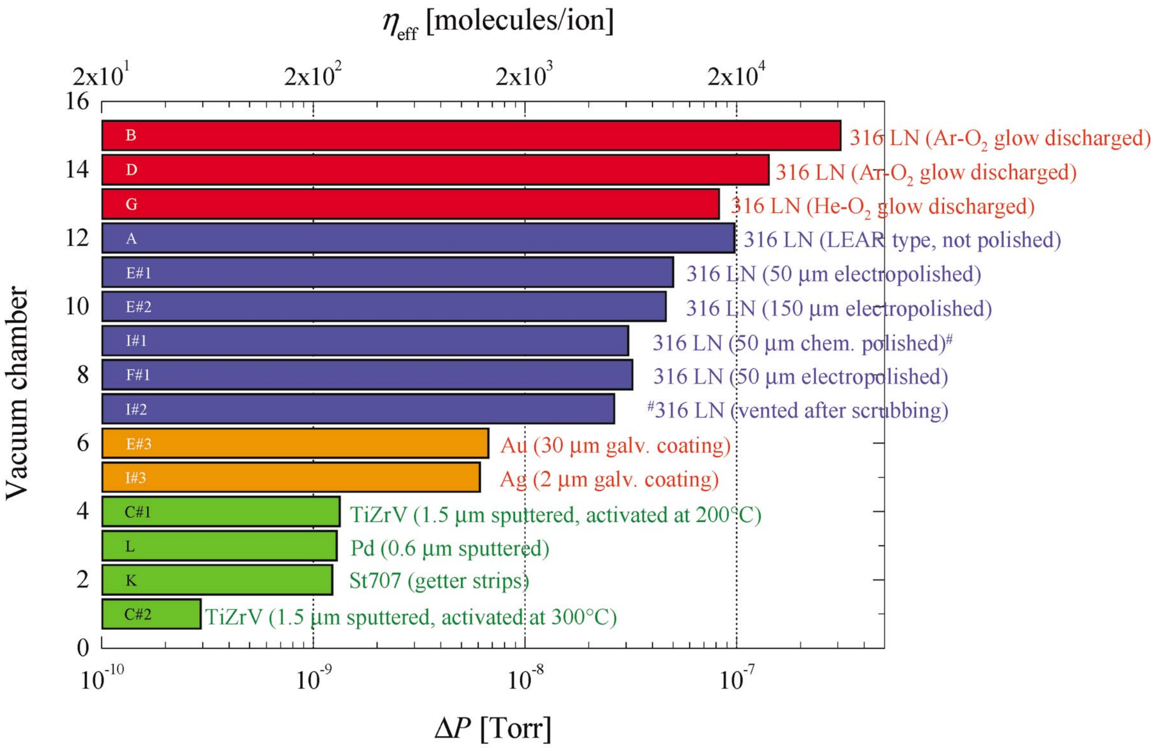

FIG. 6. (Color) Summary of pressure rise measurements for 15 different surfaces (10 different vacuum chambers) continuously bombarded with $\sim 1.5 \times 10^9$ Pb^{53+} ions under $\theta = 89.2°$ grazing incidence. The pressure increase ΔP is measured at the beginning of each scrubbing run. The plot summarizes the LINAC 3 results obtained between November 2000 and October 2002.

which both remove the surface damage layer introduced during the fabrication process of the stainless steel sheets. We conclude that there is a slight reduction in pressure rise of accelerator-type 316 LN stainless steel vacuum chambers after removal of the surface damage layer. Significant lower dynamic pressure rises were observed for all coated vacuum chambers, sputter coated getter films (Pd, TiZrV) provided the lowest dynamic gas load under heavy-ion bombardment followed by galvanic coatings (Ag, Au). The measured ΔP and corresponding η_{eff} values do not take into account the pumping speed of the chambers under test. So far, no attempt has been made to measure the intrinsic desorption yield of these films.

C. Beam scrubbing conclusions

From the above-described beam scrubbing experiments the following conclusions can be drawn from the present study:

(i) Electropolishing or chemical etching of stainless steel vacuum chambers slightly reduced the effective desorption yields.

(ii) Galvanic coatings of Ag (2 μm) and Au (30 μm) or a sputter coating of Pd (0.6 μm), performed on vacuum fired 316 LN stainless steel chambers, strongly reduced the η_{eff} values from 2×10^4 molecules/ion down to $\sim$300 molecules/ion, the lowest yield obtained for the Pd film and the St707 getter. A pumping of the coatings (Au, Ag, Pd) and the St707 getter has not been taken into consideration for the calculations of the effective desorption yields.

(iii) The following improvement factors α_i, defined as a reduced pressure increase normalized to the standard LEAR-type (not polished, not coated) 316 LN stainless steel vacuum chamber A, were obtained for the different coatings, the chemical etching, and the electropolishing:

$$\alpha_{\mathrm{epolish}} \approx 2, \qquad \alpha_{\mathrm{etch}} \approx 3, \qquad \alpha_{\mathrm{Au}} \approx 15,$$
$$\alpha_{\mathrm{Ag}} \approx 16, \qquad \alpha_{\mathrm{Pd}} \approx 76, \qquad \alpha_{\mathrm{St707}} \approx 93,$$
$$\alpha_{\mathrm{TiZrV}(200\,^\circ\mathrm{C})} \approx 72, \qquad \alpha_{\mathrm{TiZrV}(300\,^\circ\mathrm{C})} \approx 333.$$

(iv) No influence of the thickness of the noble-metal coating was observed. This fact might indicate that the desorption process is originated close to the upper surface of the coatings.

(v) No influence of the *ex situ* vacuum firing temperature (950 °C or 1050 °C) and the *in situ* bakeout temperature (300 °C or 400 °C) was measured.

(vi) The venting of a heavy-ion scrubbed stainless steel vacuum chamber resulted in the complete loss of the scrubbing effect. During the second scrubbing run, the same dose (ions/cm^2) was necessary to reduce the effective desorption yield to the value obtained before. This dedicated experiment performed at LINAC 3, which simulated for the first time a future LEIR vacuum intervention, was taken into account for the sectorization of the LEIR vacuum system.

(vii) The LEIR vacuum system will be equipped with low-outgassing absorbers, positioned at locations where increased losses of heavy ions are expected (inside the vacuum chambers of the four bending magnets). These absorbers shall be 316 LN stainless steel coated with either Au, Ag, Pd, or TiZrV. They will help to reduce the necessary LEIR scrubbing time to obtain the required dynamic gas density.

IV. SURFACE CHARACTERIZATION

A. Sample preparation

For surface analysis purposes 3 platelets (10×20 mm^2) of 316 LN stainless steel were chemically cleaned and vacuum fired at 950 °C for 2 h. Two samples were covered with Ag (~ 2 μm) and Au (~ 30 μm), both by galvanic coating on a $\sim$2 μm Ni underlayer. The third platelet was sputter coated with a $\sim$0.6 μm Pd film. A sheet of about 10×20 mm^2 was cut from the St707 getter strip and a platelet of 316 LN stainless steel was coated with TiZrV nonevaporable getter by the same procedure as for chamber C.

B. XPS surface analysis

The aim of the x-ray photoemission spectroscopy (XPS) analysis was to monitor the sample surface composition as a function of the heating time during a 300 °C bakeout in UHV and during activation in the case of the NEG coatings. The results obtained for the carbon concentration and the oxygen concentration are displayed in Figs. 7–10.

1. Silver

The as received surface showed the signals of Ag, C, O, and additionally some traces of Na and Cl. Heating up the sample to 300 °C decreased the carbon amount (see Fig. 7), but the oxygen concentration stayed constant (see Fig. 8) and Zn (~ 5 at. %) appeared on the surface. A comparison with another sample showed a correlation between the amount of Zn and O indicating that the oxygen is trapped as ZnO.

2. Gold

The as received sample presented traces of Cu in addition to Au, C, and O. After 90 min baking at 300 °C the oxygen decreased dramatically and traces of Ni appeared on the surface. Carbon remained present on the surface at a level of 25 at. %. After the bakeout the surface was considered as oxygen free.

3. Palladium

For the as received sample some Pd, C, and O were found on the surface. The level of oxygen (estimated from the OKLL line) decreased rapidly. Carbon remained at a level of 10 at. %. The Pd surface was also oxygen free after the 300 °C bakeout.

4. TiZrV and St707 nonevaporable getters

The TiZrV did not show any impurities on the surface apart from C and O. Upon heating at 200 °C in UHV the amount of O decreased rapidly (see Fig. 10), as expected for a proper activation, and reached a constant level after 6 h. A further decrease was observed by heating at 300 °C where again a constant level as a function of time was reached after a few hours. The final level of O is principally determined by the residual pressure in the analysis system, since the NEG film is active for pumping. A similar evolution was observed with carbon (see Fig. 9). The carbon present on the as received surface was constituted by airborne adsorbed hydrocarbons, as visible from the line

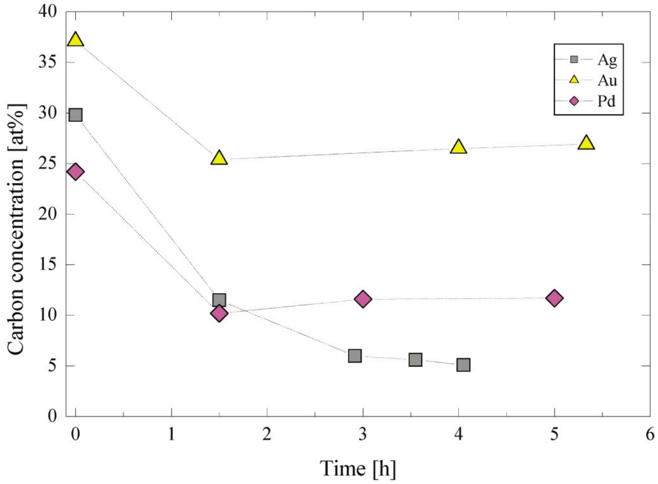

FIG. 7. (Color) Carbon surface concentration of Ag, Au, and Pd coated stainless steel during a 300 °C bakeout in UHV.

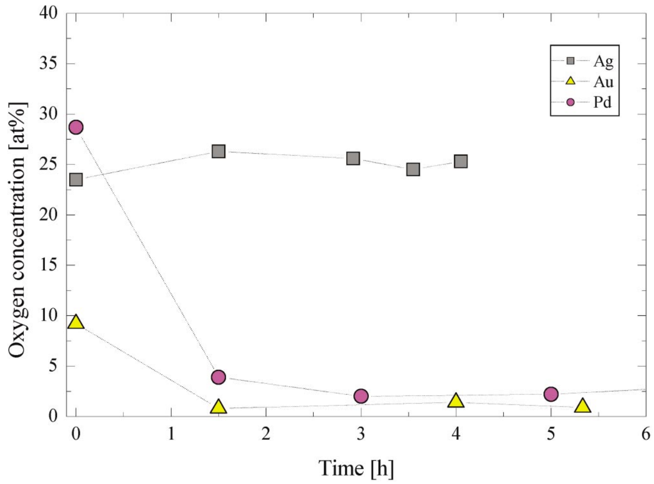

FIG. 8. (Color) Oxygen surface concentration of Ag, Au, and Pd coated stainless steel during a 300 °C bakeout in UHV.

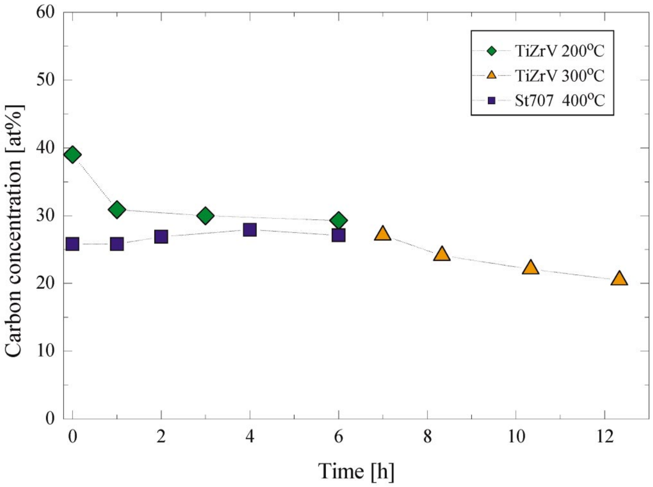

FIG. 9. (Color) Carbon surface concentration of TiZrV and St707 during a bakeout in UHV.

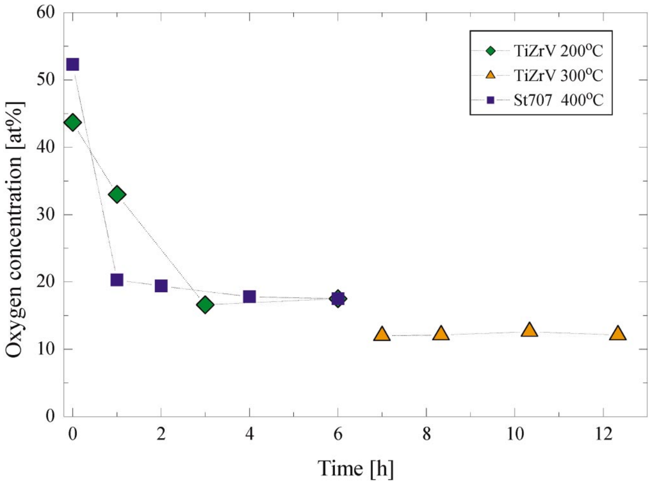

FIG. 10. (Color) Oxygen surface concentration of TiZrV and St707 during a bakeout in UHV.

shape of the C1s line (data not shown) and changed its chemical state during heating. After 200 °C activation about 60% of the detected carbon on the surface was under the chemical state of carbide and this amount increased to 90% after 300 °C. These percentages depend on the initial amount of carbon present on the surface and on the residual pressure in the analysis chamber. The carbon concentration continued to decrease even after heating several hours at 300 °C.

For St707 the activation occurs at higher temperature and therefore the sample was heated at 400 °C. The evolution is similar to that of TiZrV treated at 200 °C and also both values for C and O reached a steady state at about the same level as for the TiZrV getter with about 60% of carbon in the chemical state of carbide.

5. *Conclusion of XPS measurements*

The main contribution to the pressure increase during desorption was given by species (CO, CO_2) containing C and O. Some correlation seems to exist between the contamination level and the initial beam-induced pressure increase. The Ag and Au coated surface had approximately the same amount of C and O (30 at. % in total) after the 300 °C bakeout, the scrubbing behavior was also found to be very similar. Moreover, Pd was the surface which reached a very low level of both carbon and oxygen (14 at. % in total) after the 300 °C bakeout and exhibited also the lowest desorption rate during scrubbing. For the NEG surfaces of TiZrV (activated at 200 °C) and St707 (activated at 400 °C) the amount of C and O was almost identical as well as the corresponding initial pressure increase. The total amount of contamination was larger than for Pd, which had also the same pressure increase, but the chemical nature of carbon on Pd was quite different. Most of the carbon on the NEG was present as carbide (binding enthalpy 175 kJ/mol C for ZrC) whereas on Pd it was either adsorbed CO or graphitic carbon (CO adsorption enthalpy is 140 kJ/mol). These different species are expected to be desorbed with a quite different probability and therefore lead to a different pressure rise. A strong difference in binding enthalpy between NEG and Pd was observed also for O with about 1050 kJ/mol O_2 for ZrO and 190 kJ/mol for Pd oxide. The lowest pressure rise was obtained for the TiZrV getter activated at 300 °C, which had also the lowest amount of contamination among the analyzed NEG surfaces, with almost all the carbon under carbide form.

V. SUMMARY AND CONCLUSION

The dynamic pressure rise of accelerator-type vacuum chambers, bombarded under $\theta = 89.2°$ grazing incidence with 4.2 MeV/u Pb^{53+} ions, can be reduced at least 1 to 2 orders of magnitude by coating 316 LN stainless steel with noble metals (Au, Ag, Pd) or nonevaporable getters (TiZrV). Coated vacuum chambers and/or locally installed low-outgassing absorbers could provide a pragmatic solution to overcome severe heavy-ion-induced dynamic pressure rises currently observed in several particle accelerators. To obtain very clean surfaces after *in situ* bakeout and low dynamic gas loads under heavy-ion impact, the LEIR vacuum system will be getter coated with TiZrV wherever possible. Coated absorbers will be installed in LEIR at positions where enhanced losses are expected, namely, in the vacuum chambers of the four bending magnets.

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