Heavy-ion induced desorption yields of cryogenic surfaces bombarded 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 study the dynamic outgassing of cryogenic surfaces. Two different targets, bare and gold-coated copper, were bombarded under perpendicular impact with 4.2 MeV/u Pb^{54+} ions. Partial pressure rises of H_2, CH_4, CO, and CO_2 and effective desorption yields were measured at 300, 77, and 6.3 K using single shot and continuous ion bombardment techniques. We find that the heavy-ion-induced desorption yield is temperature dependent and investigate the influence of CO gas cryosorbed at 6.3 K. The gain in desorption yield reduction at cryogenic temperature vanishes after several monolayers of CO are cryosorbed on both targets. In this paper we describe the new cryogenic target assembly, the temperature-dependent pressure rise, desorption yield, and gas adsorption measurements.

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I. INTRODUCTION

The effect of ion-induced molecular desorption significantly influences the operation of low charge-state heavy-ion accelerators and has an important impact on the design of future machines. Since more than one decade, intense experimental studies of this phenomenon are performed in several laboratories worldwide. The results obtained were recently reviewed [1]. An important domain for further studies is the ion-induced desorption from cryogenic surfaces. With the exception of one cold-bore experiment at CERN [2], cold surfaces have not been studied using high-energy ions. Desorption experiments with cryogenic targets are motivated by heavy-ion operation of the Large Hadron Collider (LHC) at CERN as well as the conceptual and technical design of future ion accelerators, for example SIS 100, which is part of the GSI project FAIR (Facility of Antiproton and Ion Research [3]).

II. EXPERIMENTAL SETUP

A. Experimental system

The LINAC 3 experiment, used since 2000 for heavy-ion molecular desorption studies of accelerator-type vacuum chambers, has been described in full detail elsewhere [1,4]. For the desorption study of cryogenic surfaces, a cryogenic target assembly has been designed and built. The new part of the experimental system, which replaces the normally used stainless steel Conflat™ end flange of the setup, is shown in Fig. 1.

The new part of the experimental setup consists of a commercially available cold-head (Leybold Coolpower 5/100 T), various instrumentation, and a target to be tested for desorption. On the second cooling stage (6 K) of the cold-head, two resistive heaters (each 50 W, 24 V) and an intermediate gold-coated copper disk are fixed to allow for flexible target changes. Close to the target, three different ultrahigh vacuum compatible temperature sensors are mounted: a silicon diode (LakeShore DT-470), a rhodium-iron (LakeShore RF-800-4) thermometer, and a platinum resistance (PT-100) sensor. They are used for temperature monitoring during bakeout and, together with a dedicated temperature regulation system, for controlled cooldown and temperature stabilization of the target. Special care was taken for the thermal anchoring of all temperature sensors and their wires including the wires of the two heaters which were all thermalized on the first cooling stage (35 K) of the cold-head. In order to protect the cold-head assembly against the 300 K radiation of the nearby stainless steel vacuum chamber, a copper radiation shield was built and screwed onto the first stage of the cold-head. The surface of the copper shield was electropolished on its outer side and galvanically coated with black chromium inside. Prior to its installation in the LINAC 3 test setup, the cold-target assembly has been tested in the laboratory and a limit target temperature of 6.3 K was achieved.

A residual gas analyzer (Balzers QMA 125) and a Bayard-Alpert gauge (SVT 305), both calibrated, were added to the LINAC 3 test chamber which integrates the cold-target assembly. A bakable gas injection system was built and connected close to the target. The system comprises a fine dosing valve, a small vacuum chamber

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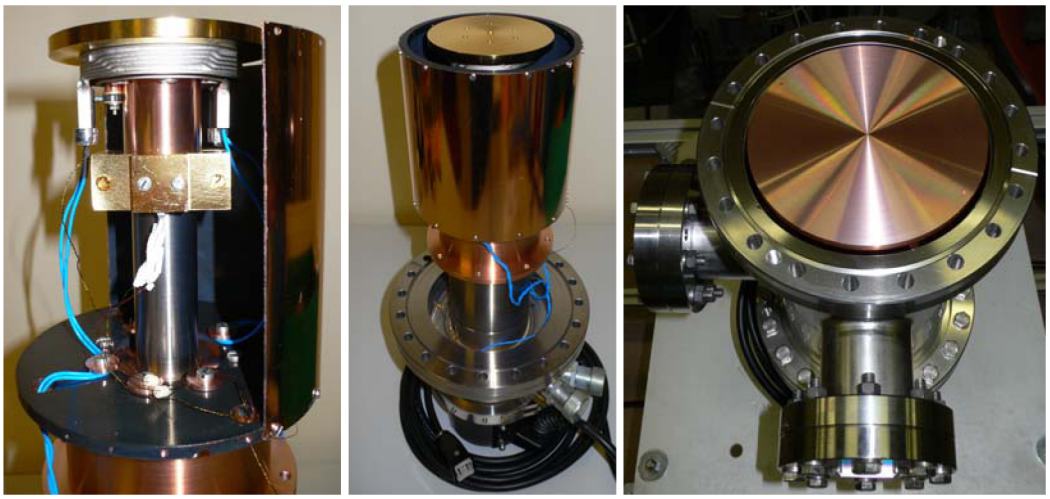

FIG. 1. Pictures of the new LINAC 3 cold-target assembly used for heavy-ion-induced desorption experiments of cryogenic surfaces. Left: cold-head with mounted heaters, temperature sensors, and partly installed thermal radiation shield (copper). Middle: cold-head with closed radiation shield. Right: assembly with mounted bare copper target integrated in a stainless steel vacuum chamber housing instrumentation feedthroughs.

with 0.5ℓ volume for the injection of a known gas quantity, two gauges for pressure control, and a turbo-molecular pumping group.

B. Fabrication and preparation of targets

Two identical oxygen-free electronic copper disks with 141 mm diameter and 6 mm thickness were manufactured and their mean surface roughness measured to be 0.17 μm. After cleaning, one of the two targets was galvanically coated with a 7 μm thin gold film and a 2 μm thin nickel underlayer, which is necessary to avoid gold-copper diffusion during bakeout of the experimental setup. In order to verify the required thickness of the nickel layer, x-ray photoemission spectroscopy measurements were performed with various samples. The results confirmed that for the chosen nickel and gold layer thicknesses no copper diffusion towards the gold surface was detectable after *in situ* bakeout at 250°C (18 h). After installation in LINAC 3, the whole experimental setup was baked at 300°C (50 h) while the bare copper target was kept at 150°C (92 h). For the second ion desorption test of the gold-coated copper target the same bakeout procedure was applied.

C. Experimental procedure

As already mentioned before, a gold-coated and a bare copper target were tested for desorption and effective ion-induced desorption yields have been measured for a fixed impact angle of $\Theta = 0°$ (perpendicular) using 4.2 MeV/u Pb^{54+} ions. Experiments were performed at 300, 77, and 6.3 K target temperatures. For determination of the desorption yields the pressure rise method was applied, which is described in detail elsewhere [4]. The effective desorption yield η_{eff} (molecules/ion) is 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 ΔP is the total pressure increase during continuous bombardment, S is the pumping speed in ℓ/s, $\dot{N}_{\mathrm{Pb}}$ is the number of impacting lead ions per second, k_B is the Boltzmann constant, T is the temperature of the released gas (300 K) at the position of the vacuum gauge, and G is a constant that converts gas quantities (Torr $\times$ ℓ) into number of molecules ($G \approx 3.2 \times 10^{19}$ at 300 K).

D. Pumping speed of a cryogenic target

In comparison to measurements with targets at ambient temperature, the total pumping speed S of the test setup increases for targets cooled down to cryogenic temperatures, we define

$$S = S_{\mathrm{at}} + S_{\mathrm{cryo}},$$

where S_{at} is the ambient temperature pumping speed of the system, given by the installed sputter-ion and titanium-sublimation pumps. S_{cryo} is the additional pumping speed given by the surface area of a cryogenic target.

We have chosen the following experimental approach to determine S as a function of the target temperature: by bombarding the sample with a single shot (620 μs long) of about 1.3×10^9 Pb^{54+} ions, the partial pressure rise and characteristic decay time can be measured for H_2, CH_4, CO, and CO_2 using the calibrated residual gas analyzer. The gas specific pressure decay times are fitted and pumping speeds S are determined for all investigated target temperatures. This single shot measurement technique and the pumping speed evaluation have been described in detail elsewhere [4].

In a first step of each experiment, the total pumping speed of the system has been measured at 300, 77, and 6.3 K. Second, the investigated sample was continuously bombarded for about 1 h with lead ions to determine the desorption yield. One hour was chosen to avoid a potential projectile-induced cleaning (''beam scrubbing'') of the bombarded surface. At the lowest achievable target temperature of 6.3 K, the baked gas injection system was used to inject and cryosorb several monolayers (ML) of CO onto the cold surface. After each CO injection, up to a maximum of 300 ML, heavy-ion-induced desorption yields were measured at 6.3 K using the pressure rise method.

III. RESULTS

A. Desorption yield measurements at 300 K

The bare and the gold-coated copper targets were continuously (every 1.2 s) bombarded with 4.2 MeV/u Pb^{54+} ions under perpendicular incidence ($\Theta = 0°$). The pressure rises, measured with the residual gas analyzer (RGA) and the Bayard-Alpert gauge (BAG), are displayed in Fig. 2. One can clearly see that the ion-induced pressure rise of both targets is dominated by desorption of CO, followed by H_2, CO_2, and CH_4. As described above, the pumping speeds were evaluated with the single shot technique. For both targets, $S \approx 15\ \ell/s$ (N_2 equivalent) was determined. Together with the measured total pressure rise the corresponding effective 300 K desorption yields are derived and displayed in Fig. 2. We find $\eta_{eff} \approx 8100$ molecules/ion for the bare copper and $\eta_{eff} \approx 6200$ molecules/ion for the gold-coated copper target.

B. Desorption yield measurements at 77 K

In the second step of each experiment, both targets were cooled down from 300 to 77 K and the sample temperature was stabilized using the regulation system comprising the

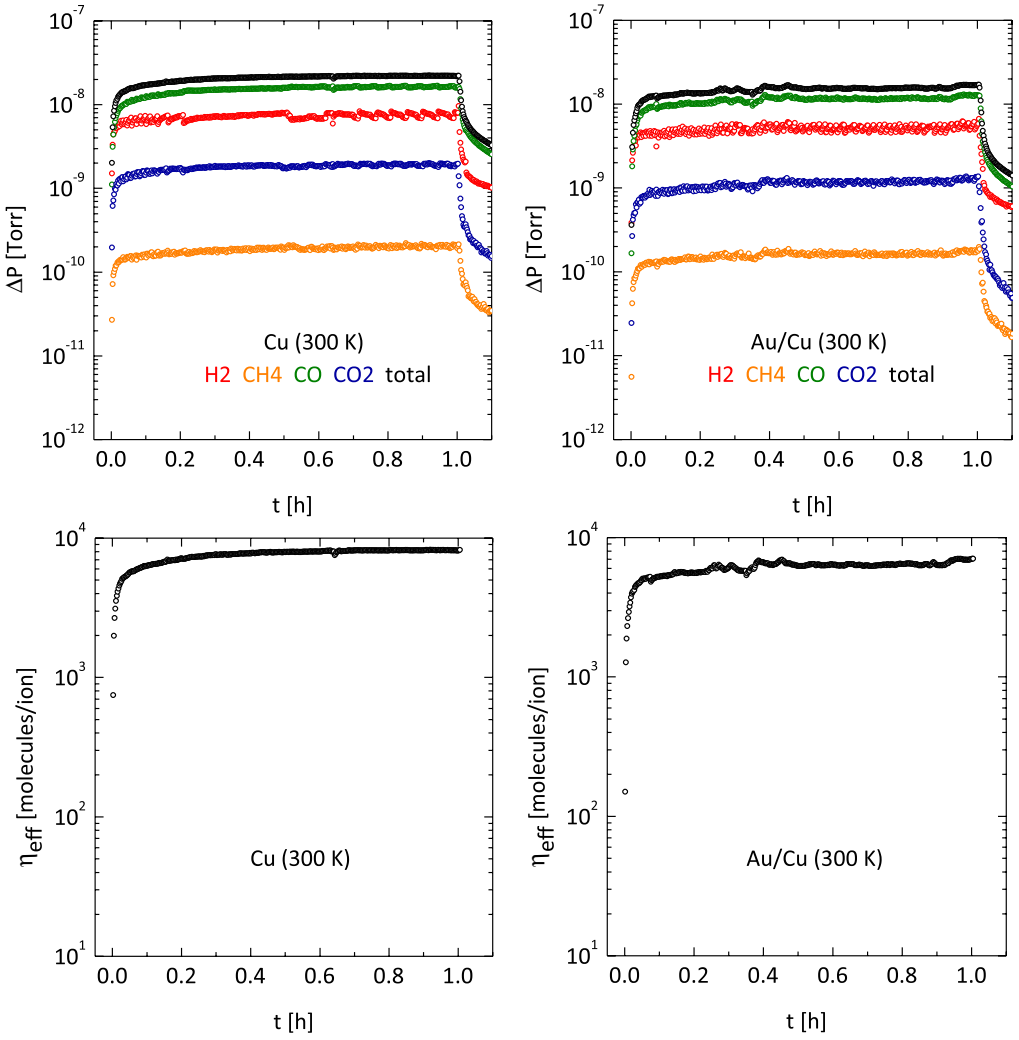

FIG. 2. Heavy-ion-induced gas desorption measurements at 300 K target temperature. Partial pressure rises (ΔP) of H_2, CH_4, CO, CO_2, and total pressure measured for bare and gold-coated copper samples continuously bombarded under $\Theta = 0°$ with 4.2 MeV/u Pb^{54+} ions are shown together with the corresponding desorption yields (η_{eff}).

DC heaters and the Si diode sensor, both mounted on the back side of the sample support disk. The measured pumping speeds (N_2 equivalent) were evaluated with single ion shots to be $S_{Cu} \approx 118\ \ell/s$ and $S_{Au/Cu} \approx 83\ \ell/s$. The obtained results for the 77 K partial pressure rises and the corresponding desorption yields are summarized in Fig. 3. The heavy-ion-induced pressure rise is dominated by H_2, which is about 1 order of magnitude larger than for CO, followed by CO_2 and CH_4. We find $\eta_{eff} \approx 2900$ molecules/ion for the bare copper and $\eta_{eff} \approx 1600$ molecules/ion for the gold-coated copper target.

It is worthwhile to mention that the RGA partial pressure signals fluctuate more at 77 K compared to 300 K. This can be explained by the very low partial pressures measured with the RGA and the additional pumping speed of the cryogenic target. The ΔP oscillations, visible in Fig. 3, are more pronounced for CH_4 and CO_2, for which the partial pressure values are in the 10^{-12} Torr range. For CO and H_2, the measured partial pressures at 77 K are in the 10^{-10} Torr and 10^{-9} Torr ranges where the RGA signals are more stable. Since the ion bombardment rate, which is one shot every 1.2 s, is not synchronized with the RGA data acquisition rate (one measurement every 7 s), this yields to a pressure rise after each shot which is subsequently pumped before the next ion bombardment of the cold target. This pumping speed effect has been clearly demonstrated in the past by bombarding a TiZrV getter-coated stainless steel vacuum chamber in the same experimental setup [4].

C. Desorption yield measurements at 6.3 K

After the 77 K desorption experiments both samples were cooled down to 6.3 K, which is the limit temperature of the cold-head assembly. Again, single lead-ion shots were used to determine the total pumping speed (N_2 equivalent) of the setup resulting in $S_{Cu} \approx 215\ \ell/s$

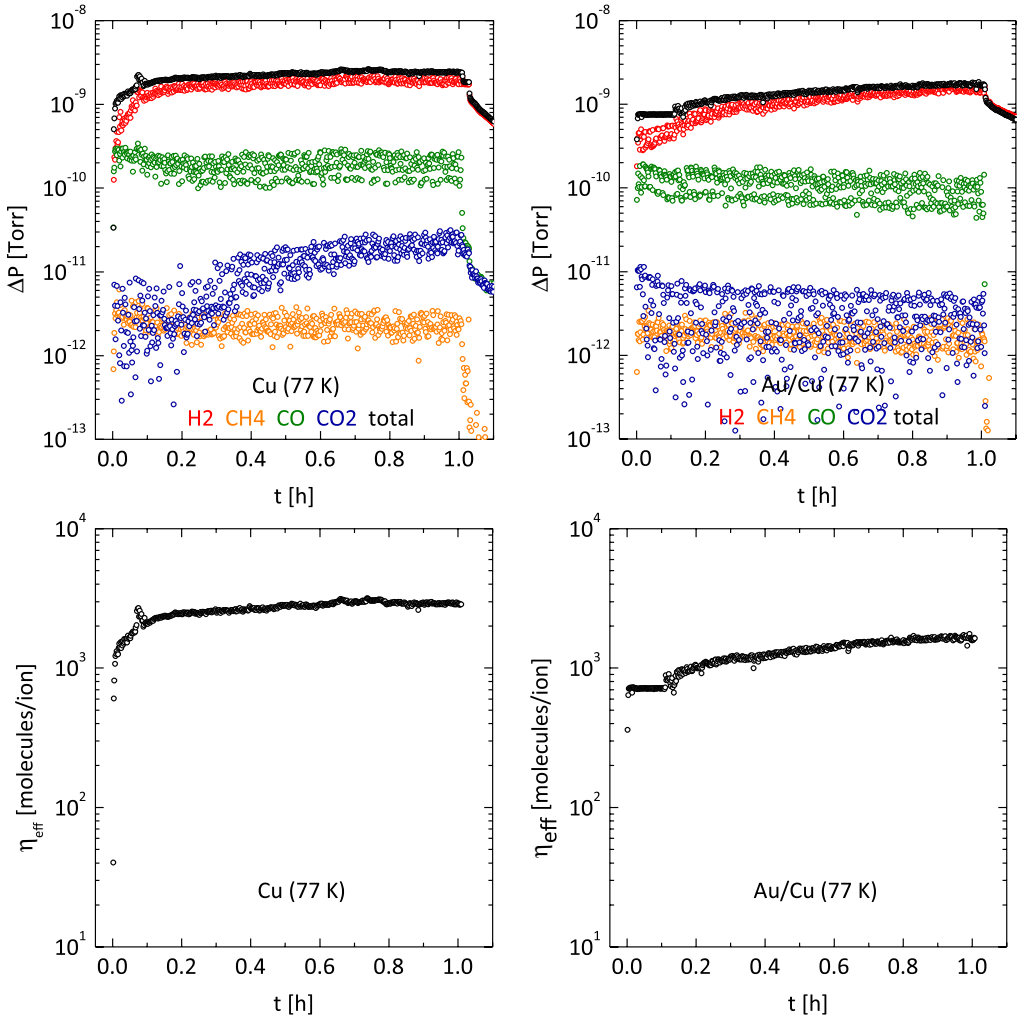

FIG. 3. Heavy-ion-induced gas desorption measurements at 77 K target temperature. Partial pressure rises (ΔP) of H_2, CH_4, CO, CO_2 and total pressure measured for bare and gold-coated copper samples continuously bombarded under $\Theta = 0°$ with 4.2 MeV/u Pb^{54+} ions are shown together with the corresponding desorption yields (η_{eff}).

and $S_{Au/Cu} \approx 209\ \ell/s$ at 6.3 K. The measured partial pressure rises and ion desorption yields are displayed in Fig. 4. As already observed at 77 K, gas desorption is dominated by H_2 and CO molecules. At 6.3 K the desorption yields are $\eta_{eff} \approx 460$ molecules/ion (copper) and $\eta_{eff} \approx 340$ molecules/ion (gold-coated copper). Due to the low partial pressures and the high additional pumping speed of the cryogenic targets, a rather strong oscillation of the RGA signals, as already explained before, is also observed at 6.3 K.

D. Temperature dependence of the yield

The above described experiments have shown, for the first time, that the heavy-ion-induced desorption yield is temperature dependent. The obtained results are summarized in Fig. 5. Bare and gold-coated copper surfaces, bombarded under perpendicular impact with 4.2 MeV/u Pb^{54+} ions, both have a factor of ≈ 18 lower desorption yield at 6.3 K compared to the measured 300 K value.

E. CO gas adsorption experiments at 6.3 K

The temperature-dependent desorption experiments described so far were conducted in a way that a minimum of gas could be adsorbed on both targets prior to their cooldown to cryogenic temperatures. The aim of the gas adsorption experiments was to test if and how much the ion-induced desorption yield changes with the amount of gas adsorbed at low temperature. Apart from the fundamental aspect of such a study, we consider that it also has some relevant practical applications for the design and operation of cold vacuum systems, either for existing or future heavy-ion accelerators.

The baked gas injection system was used for the injection of CO onto the cryogenic target surface. The experimental procedure applied for both targets was as follows. First, CO

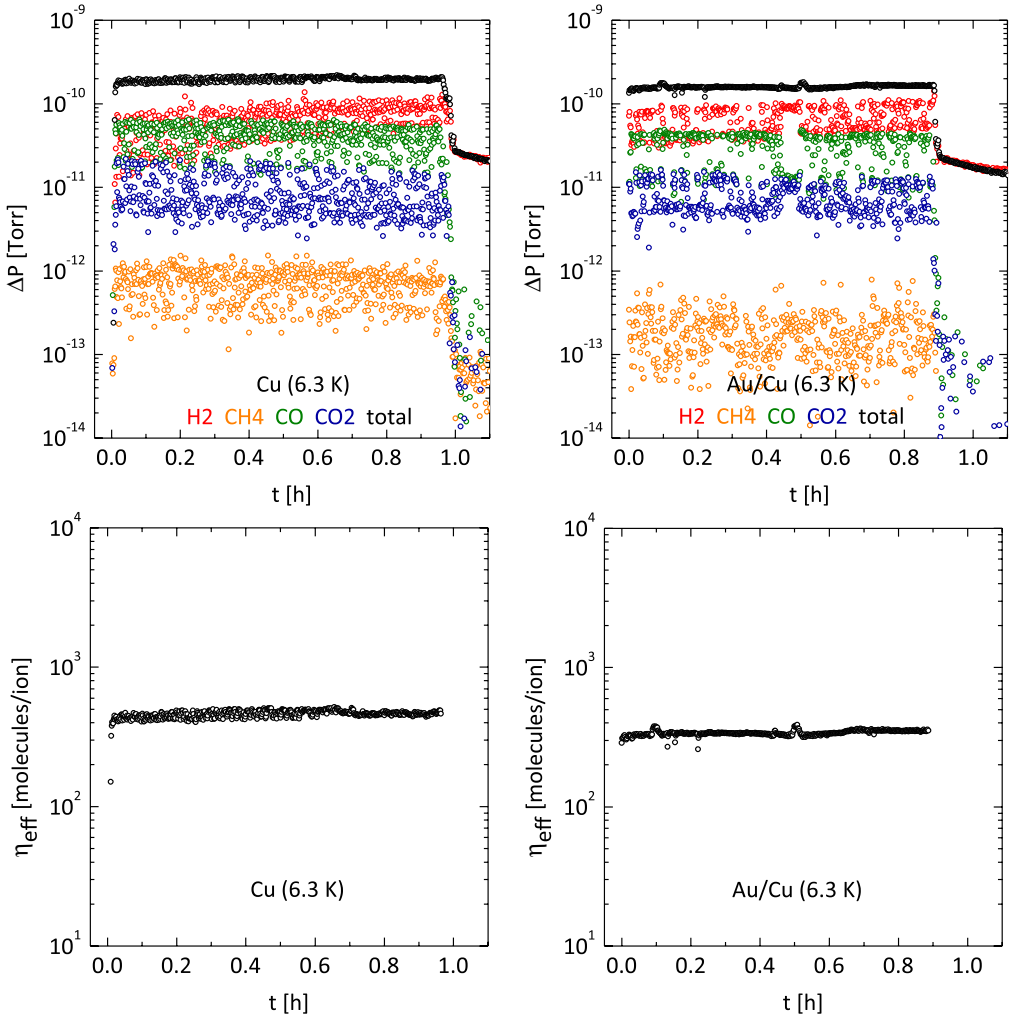

FIG. 4. Heavy-ion-induced gas desorption measurements at 6.3 K target temperature. Partial pressure rises (ΔP) of H_2, CH_4, CO, CO_2 and total pressure measured for bare and gold-coated copper samples continuously bombarded under $\Theta = 0°$ with 4.2 MeV/u Pb^{54+} ions are shown together with the corresponding desorption yields (η_{eff}).

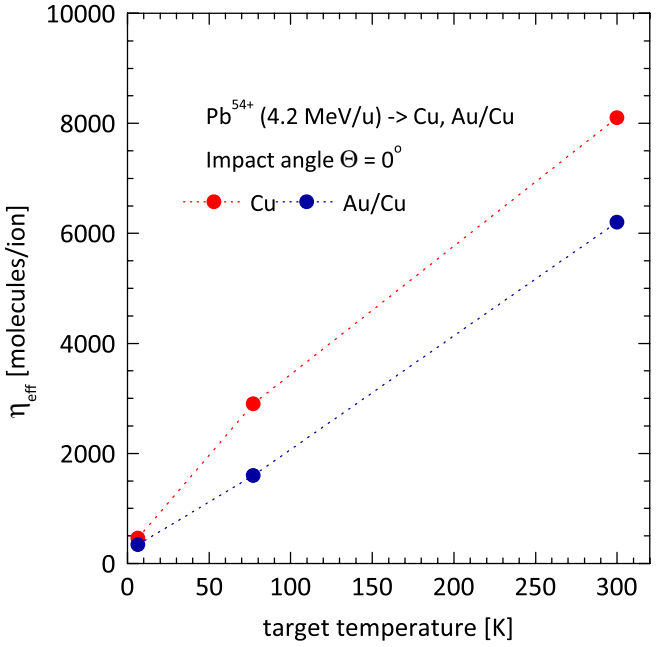

FIG. 5. Temperature dependence of the heavy-ion-induced desorption yield of bare and gold-coated surfaces bombarded under $\Theta = 0°$ with 4.2 MeV/u Pb^{54+} ions.

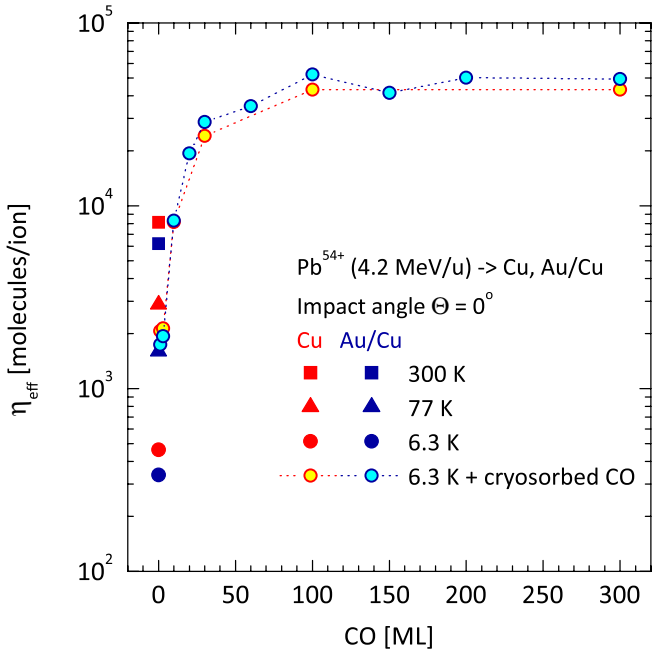

FIG. 6. Dependence of the heavy-ion-induced desorption yields on CO gas adsorption. Full symbols: 300, 77, and 6 K yields without gas injection. Open symbols: yields measured at 6.3 K as a function of cryosorbed CO monolayers.

gas was injected into the vacuum system and a known amount of monolayers adsorbed on each sample. Second, the injected gas was pumped out until the limit pressure of the system was reached again. Afterwards, the targets were bombarded for 1 h with Pb^{54+} ions under perpendicular impact and the pressure rise was measured with the RGA and BAG. After this continuous ion bombardment and as soon as the limit pressure was reached again, the procedure was continued with the first step, the cryosorption of more CO monolayers on each target. The results of 16 such measurements, performed with different quantity of CO gas adsorbed at 6.3 K, are summarized in Fig. 6.

For both cryogenic targets, the 6.3 K heavy-ion-induced desorption yields were lowest without additional gas adsorption, desorption rates of about 400 molecules/ion were measured. The yields increased by a factor of ≈ 5 to about 2000 molecules/ion, after the adsorption of 1 ML of CO gas onto the cold surfaces. A further injection of 3 ML did not change desorption rates significantly. We note that the yield measured at 6.3 K with 3 ML of CO equals with the 77 K result obtained without CO adsorption. After 10 ML of CO were cryosorbed at 6.3 K, a yield of ≈ 8200 molecules/ion was measured for both targets. This value is identical with the yield found for the bare copper target at ambient temperature. As can be seen from Fig. 6, the η_{eff} values increase to about 5×10^4 molecules/ion after 100 ML and remain constant until 300 ML of CO gas adsorbed at 6.3 K. We further point out that the low-temperature heavy-ion-induced desorption rate, measured as a function of adsorbed CO quantity, is independent of the target material, i.e., copper or gold.

IV. SUMMARY AND CONCLUSION

The dynamic pressure rise of accelerator-type targets, bombarded under $\Theta = 0°$ perpendicular incidence with 4.2 MeV/u Pb^{54+} ions was studied at 300, 77, and 6.3 K. The heavy-ion-induced desorption yields of both targets, bare and gold-coated copper, were reduced by a factor of about 18 to η_{eff} (6.3 K) $\approx 400 \pm 60$ molecules/ion, compared to ambient temperature. The gas desorption of both cryogenic surfaces was dominated at 6.3 K by hydrogen. The adsorption of several monolayers of CO increases the low-temperature yields significantly up to an equilibrium value of η_{eff} (6.3 K) $\approx 5 \times 10^4$ molecules/ion, measured for the gold-coated copper target, which is 8 times larger than the desorption yield obtained at ambient target temperature without CO adsorption.

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