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ELECTRON STIMULATED DESORPTION FROM CRYOGENIC NEG-COATED SURFACES

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Abstract

Non-Evaporable Getter (NEG) coating has been used for years in many particle accelerators due to its advantages like evenly distributed pumping speed, low thermal outgassing, and low photon, electron and ion stimulated desorption yields. Although NEG coating has been tested at room temperatures intensively, there is little data on its behaviour at cryogenic temperatures. Tests in this environment are important for the Future Circular Collider (FCC) study and other accelerator facilities where the operational conditions of the beam screen are restricted to cryogenic temperatures. This work will provide some preliminary results on NEG properties at low temperatures, e.g. pumping speed and capacity, as well as its behaviour under electron bombardment, where electron stimulated desorption (ESD) yields will be calculated. The ternary Ti-Zr-V coating, deposited with dense and columnar structure, will be the first material to be tested at cryogenic temperatures in ASTeC Daresbury laboratory. The results were compared with the ones obtained at room temperature, offering an insight into the behaviour of NEG-coated cryogenic chambers when beam-induced effects are present.

INTRODUCTION

The non-evaporable getter (NEG) coating was developed at CERN [1-5] and since has become an important vacuum technology. Not only does it provide distributed pumping but also reduces the photon and electron stimulated desorption (PSD and ESD) yields [6-8], which allows reduction of the number of pumps and their size (i.e. pumping speed). It is often the only way to ensure that required UHV conditions are reached in long and narrow vacuum chambers in particle accelerators. However, a beam vacuum pipe of the Future Circular Collider (FCC) would be exposed to conditions that have not yet existed in any accelerators before. Not only will the beam pipe have a large aspect ratio, and hence limited vacuum conductance, but also it will be placed inside a 1.9 to 4.5 K coldbore. The temperature of the beam pipe will be in the range of 20 to 40 K [9]. Since the vacuum conductance is proportional to $\sqrt{T}$, the distributed pumping created with NEG coating could play an important role in ensuring UHV conditions in the beam pipe. However, the behaviour of the NEG coating at low temperatures has not been extensively investigated. [10]. In this study, two Ti-Zr-V samples were tested at various temperatures in order to study how low temperatures influence sticking probability of the coating and ESD yields from it.

DEPOSITION

Two samples were deposited in ASTeC from a Ti-Zr-V target provided by NSRL, USTC (China). This specific composition of the coating was chosen due to a larger amount of results on pumping properties and ESD at room temperature (RT) being available [6-8] and refs. within]. The identical substrates were 316LN stainless steel tubes with a length of 50 cm and an inner diameter of 3.8 cm equipped with CF40 flanges on both ends. The deposition facility has been described in ref. [11]. A Ti-Zr-V target, with a chemical composition (atomic percentage) of Ti:Zr:V roughly being 1:1:1, in a form of a 3-mm diameter alloy wire was used for the deposition. Both substrates were prepared and deposited under the same conditions. After installing the sample substrate, the deposition facility was baked at 150 °C for four days. After cooling down, the pressure in the system was approximately 2×10^{-9} mbar. The deposition was performed with Kr as a discharge gas at 0.1 mbar with a DC power supply, at the substrate temperature in the range of 90-110 °C. Deposition parameters were selected (from previous experience) to result in a columnar structure of NEG coating.

EXPERIMENTAL FACILITY

The samples were vented to air and transferred to the NEG-coating characterisation facility [11-13] located at the same laboratory and experiments performed.

Sample 1 was used to study the H2 and CO initial sticking probabilities and CO pumping capacity of the NEG material in RT. Gases were injected in to the system through a gas injection line at the bottom of the NEG coated tube. During the gas injection, the valves to the other pumps were closed. Partial pressures were measured at the top and bottom of the tube by using residual gas analysers (RGAs). The ratios of partial gas pressures were calculated and used for obtaining the sticking probability and pumping capacity.

Sample 2 was to be installed on another testing chamber allowing control of the sample temperature in the range of 90-300 K with liquid nitrogen (LN2) flowing through a sample chiller. Kooltherm® phenolic pipe insulation was used to cover the chiller, the tube and bellows at either side. The other parts of the test facility remained at RT. This system allowed ESD measurements using a Thoria coated

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filament stretched along the sample as a source of electrons, as described in Ref. [8].

EXPERIMENTAL PROCEDURE AND RESULTS

A standard ASTeC NEG activation procedure has been applied to activate Sample 1 to 120, 140, 150, 160, 180, 200, 220, 250 and 300 °C for 24 hours [12]. The results of Sample 1 measurements at RT were used to obtain the H_2 and CO initial sticking probabilities and CO pumping capacity as a function of activation temperature. The sticking probabilities were obtained from partial pressure ratios with the results of test particle Monte Carlo modelling [13] using Molflow software [14]. The pumping capacity corresponds to the amount of injected gas normalised to NEG coating surface area. The Sample 1 results show that maximum CO pumping is reached at 140 °C, while H_2 pumping gradually increases, see Fig. 1.

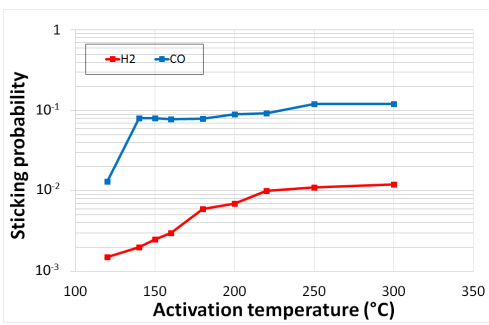

Figure 1: H_2 and CO sticking probability of sample 1 as a function of activation temperature.

Sample 2 was installed on the second Testing Chamber (TC) and underwent a different experimental procedure.

The TC was baked out to 200 °C for 24 hours, keeping the NEG-coated parts at approximately 80 °C. Then it was cooled down to 150 °C. RGAs, extractor gauges and the ESD filament were switched on and degassed; note that all hot filaments remained switched on until the end of experiment. Then the TC and the sample were cooled down to RT and kept at RT for 3 days, until the pressure in the test chamber reached 1.5×10^{-8} mbar.

Run 1

The ESD measurements from non-activated NEG coating were started at RT, then the sample was cooled down to 90 K, then warmed up to RT while the electron bombardment was still on.

The partial pressures for different gas species measured with top RGA and a sample temperature are shown in Fig. 2 as a function of accumulated electron dose. One can see that pressures reduced with temperature, however, for the accelerator vacuum system design the most interesting value would be gas density inside the cryogenic sample tube. The pressure measured by the RGA inside at RT port connected to a sample tube is:

$$P_{RGA} = n_{RGA} k_B T_{RT}. \quad (1)$$

Two gas densities inside the RGA and the sample tube at different temperatures are related as:

$$n_{RGA}\overline{v_{RT}} = n_t \bar{v}_t, \quad (2)$$

where $\bar{v} = \sqrt{\frac{2k_B T}{\pi m}}$ is the mean molecular velocity. Therefore, gas density inside the sample tube shown in Fig. 2 has been calculated with:

$$n_t = n_{RGA}\frac{\overline{v_{RT}}}{\bar{v}_t} = \frac{P_{RGA}}{k_B\sqrt{T_{RT}T_t}} \quad (3)$$

The initial ESD yields shown in Table 1 were calculated only for the results obtained at RT because the sticking probabilities of non-activated NEG coating at the beginning of the electron bombardment were equal to zero; however, they are not defined as a function of temperature and electron dose.

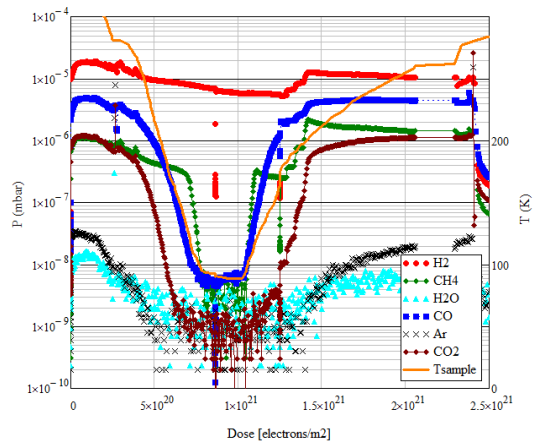

Figure 2: Partial pressures as a function of electron dose for sample 2 in Run 1.

Table 1: Initial ESD Yields, η (Molecules/e-), at RT Obtained from Sample 2 in Run 1 (Non-activated) and Run 2 (activated at 180 °C)

Gas	H_2	CH_4	CO	Ar	CO_2
Run 1	0.2	6×10^{-3}	0.025	2×10^{-4}	6×10^{-3}
Run 2	0.01	2×10^{-3}	5×10^{-3}	2×10^{-6}	1.5×10^{-3}

Run 2

The NEG coating was activated to 180 °C for 24 hours then cooled down to RT. The ESD measurements were started at RT for 1-2 hours. Then electron bombardment was interrupted to install the sample chiller. The ESD measurements started again at RT, and the sample was cooled down to and bombarded at 90 K for 3 hours, then warmed up to RT, see Fig. 3 for partial pressures.

H_2 and CO sticking probabilities at RT were measured by short injection (to avoid NEG saturation) of these two gases, as described for Sample 1 above. Sample 2 sticking probabilities were found to be $\alpha_{H2} = 0.005$ and $\alpha_{CO} = 0.05$. These values are comparable to the ones for Sample 1 also activated to 180 °C (see Fig. 1): $\alpha_{H2} = 0.006$ and $\alpha_{CO} = 0.075$.

Sticking probabilities as a function of temperature were measured with the use of thermal outgassing from the ESD filament. The temperature of the filament was increased by putting a higher current than in a normal operation. The measurements started at RT then the sample was cooled

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down to and bombarded at 90 K while the TC remained pumping. Then the valve to the pump was closed and the samples were warmed up. At the end of the run the temperature of the filament was set back to normal.

The calculations of sticking probability at cryogenic temperatures can be done considering that distributed thermal outgassing along the sample is q, when the gas density n_t inside an infinity long tube with sticking probability α is $n_t = \frac{4q}{\alpha \overline{v_t}}$. Hence, using Eqs. (1)-(2):

$$P_{RGA} = k_B T_{RT} n_t \frac{\overline{v_t}}{\overline{v_{RT}}} = k_B T_{RT} \frac{4q}{\alpha \overline{v_{RT}}}, \quad (4)$$

Therefore, partial pressure is inversely proportional to sticking probability; hence the sticking probability at cryogenic temperature αCT can be calculated from known sticking probability at RT, αRT, as follows:

$$\alpha_{CT} = \alpha_{RT} \frac{P_{RT}}{P_{CT}} \quad (5)$$

The calculated sticking probability as a function of temperature is shown in Fig. 4, showing that sticking probability increases with decreasing temperature. The obtained $\alpha_{H2}(T)$ and $\alpha_{CO}(T)$ will be used to calculate the ESD yields at RT and cryogenic temperatures.

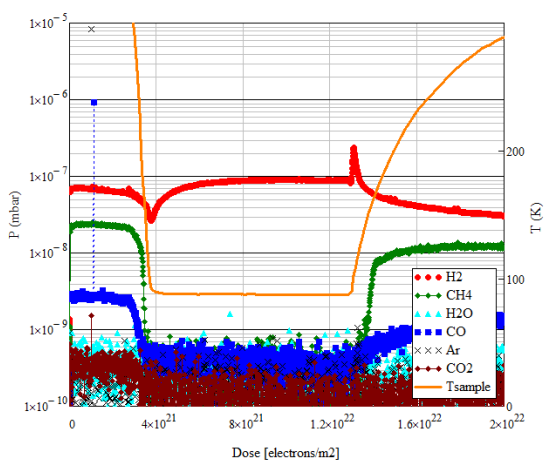

Figure 3: Partial pressures as a function of dose for sample 2 in run 2.

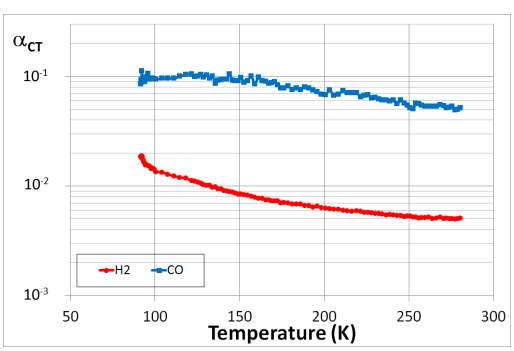

Figure 4: H_2 and CO sticking probabilities, α_{CT}, as a function of sample 2 temperature in Run 2.

DISCUSSION

The Ti-Zr-V coating has sticking probabilities of H_2 and CO reported previously at around 0.005-0.01 and 0.1-0.6, respectively [3, 8, 11]. The H_2 and CO sticking probabilities shown for Sample 1 in Fig. 1 are similar to our earlier results. The surface characterisation of these samples is still ongoing.

Using Sample 2, which must be identical to Sample 1 after deposition but before pumping experiments, enables evaluating the effect of cryogenic temperatures on ESD and sticking probabilities.

In Run 1, shown in Fig. 2, the partial pressures measured with the top RGA for different gas species went down with a sample temperature. In general, this effect could be explained by both ESD and sticking probability dependence on temperature and as well as a sample conditioning with electron dose. Since the measurements were performed during cooling down and warming up and show the same trend, the latter can be excluded. However, it does not allow separation of the ESD and sticking probability dependence on temperature.

In Run 2, $\alpha_{H2}(T)$ and $\alpha_{CO}(T)$ were measured, see Fig. 4. If at room temperature the pumping is provided by chemisorption of NEG coating, then at lower temperature it has an additional contribution of cryosorption, resulting in increasing α_{H2} by a factor of ~3.5 and α_{CO} by a factor of ~2 at T = 90 K. The results for H_2 are practically the same as reported in [10]. No results were previously published on CO.

Table 1 shows that after activation the ESD yields reduced by a factor of ~20 for H_2, ~5 for CO, ~4 for CO_2, ~3 for CH_4 and ~100 for Ar.

When cooling was started, the gas density for all gases reduced significantly. The ESD yields as a function of temperature should be evaluated in future.

CONCLUSIONS AND FUTURE PLANS

Pumping and ESD properties of columnar Ti-Zr-V coating were measured between 90 and 312 K. The gas density in the vacuum chamber bombarded with electrons reduced with temperature for the NEG coating which was either not activated or activated to 180 °C. The sticking probability of H_2 and CO for NEG coating activated to 180 °C increased when the sample was cooled down.

The study will continue for NEG coating activated to higher temperature and various film structures and morphologies.

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