# FURTHER OPTIMISATION OF NEG COATINGS FOR ACCELERATOR BEAM CHAMBER *

O.B. Malyshev# and R. Valizadeh, ASTeC, STFC Daresbury Laboratory, Warrington, UK

*Abstract*

The non-evaporable getter (NEG) coating, invented at CERN in 90s, is used nowadays in many accelerators around the world. The main advantages of using NEG coatings are evenly distributed pumping speed, low thermal outgassing rates and low photon and electron stimulated gas desorption. The only downside of the NEG is its selective pumping: it pumps $H_2$, CO, $CO_2$ and some other gas species, but does not pump noble gases and hydrocarbons. However, in the accelerators where NEG coating could be beneficial, there is synchrotron radiation and photoelectrons that bombard vacuum chamber walls, it was found in our study that hydrocarbons can be pumped by NEG coating under electron and, most likely, photon bombardment. The detail and the results of this study are reported in this paper.

## INTRODUCTION

Since its invention [1] the non-evaporable getter (NEG) coating was applied in vacuum systems of many accelerators around the world, mainly in SR sources (ESRF, ELETTRA, Diamond LS, Soleil, MAX-IV, etc.) and in colliders (LHC, ILC, etc.). The combination of NEG coating advantages, such as evenly distributed pumping speed, low thermal outgassing rates and low photon and electron stimulated gas desorption, allows building a vacuum system that either could not be realised without NEG coating or allows to significantly reduce the cost of UHV vacuum system. The optimisation of NEG coating was focused in ASTeC in a few directions: lowering NEG activation temperature to 150 ºC was achieved by using a quaternary Ti-Zr-Hf-V layer [2,3] as compared to 180 ºC for CERN's ternary Ti-Zr-V layer [4,5]; lowering the photon and electron stimulated gas desorption yields [6,7] was achieved by having a denser layer at the interface [8]. It was also found that the non-activated or CO saturated NEG coating can be activated by synchrotron radiation (SR) or electron bombardment. The selective pumping of NEG coating is a performance limiting factor, NEG pumps $H_2$, $H_2O$, CO, $CO_2$ and some other gas species but does not pump noble gases and hydrocarbons. If the vacuum system has no leaks to air and no gas injection, then the amount of noble gases should be negligible. Contrary to that, there is very little hydrocarbons in air or in a conventional vacuum chamber, but they (mainly $CH_4$) are generated on the NEG surface during the absorption process of $H_2$, $H_2O$, CO and $CO_2$ [6]. It is also known that the same NEG can pump hydrocarbons at higher temperature [9] and that the pumping speed of a sputter ion pumps for $CH_4$ increases in the presence of NEG [10]. The aim of this reported work was to investigate the $CH_4$ pumping properties of NEG coating.

#oleg.malyshev@stfc.ac.uk

## ESD FACILITY

A dedicated facility for studying NEG coating properties such as sticking probability, pumping capacity and electron stimulated desorption (ESD) is described details in [7] and is shown schematically in Fig. 1. Tubular samples are quite a convenient geometry which allows avoiding NEG coating poisoning by gas desorbed from non-coated parts of the facility during NEG activation [11]. Electrons emitted from a filament running along the centre of a tubular sample bombard its inner surface with electron energies up to 6.5 keV and a current up to 200 mA. The sample temperature can be controlled with a cooling jacket in the range between -15 and +80 ºC; in reported experiments this was set to 30 ºC. Pressures $P_1$ and $P_2$ are measured with Residual Gas Analysers (RGA) as well as with an extractor gauge in the test chamber.

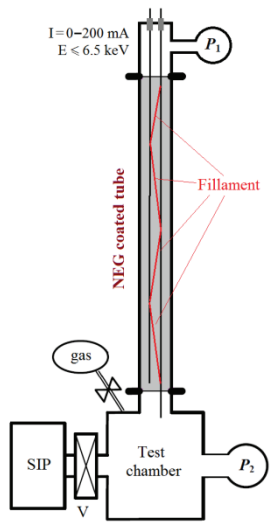


Figure 1: Schematic layout of ESD facility.

## SAMPLE PREPARATION

A 0.5-m long and 38-mm diameter 316LN stainless tube was used as a sample in this experiment. This sample was used in other previous experiments. Initially the sample was installed on the ESD facility and baked in-situ to 250 ºC for 24 hours, bombarded with 500-eV electrons reaching a similar accumulated dose and ESD yields. It was then vacuum fired to 950 ºC for two hours at a pressure of around $10^{-5}$ mbar and again installed on the ESD facility, baked in-situ to 250 ºC for 24 hours, bombarded with 500-eV electrons reaching a similar accumulated dose and ESD yields, as reported in [12].




ISBN 978-3-95450-132-8

Then it was coated with a dense Ti-Zr-Hf-V NEG film and installed at the ESD facility to perform the NEG activation at 150 ºC, followed by ESD yield measurements, $H_2$ and CO sticking probability measurements and CO saturation. The measurements were repeated after NEG activations at 180, 250 and 350 ºC [13]. The experiments described below were performed between the ESD yield measurements with NEG coating activated at 350 ºC and before the sticking probability measurements.

## RESULTS AND DISCUSSIONS

### *Initial Conditions*

The total pressure measured with an extractor gauge without electron bombardment was ~$7\times10^{-11}$ mbar with only hydrogen detectable with an RGA, with $P_2 \approx 30\ P_1$. Closing a valve (V on Fig.1) to the sputter ion pimp (SIP) does not affect these values. In Fig. 2 (and the following figures) and the following figures the $P_1$ partial pressures of detectable gas species are shown. The following parameters were varied during the experiment (see Table 1):

- external pumping speed by opening or closing the valve V between the test chamber and SIP;
- injected gas flow;
- electron energy;
- switching on and off the electron beam;
- switching on and off the filament.

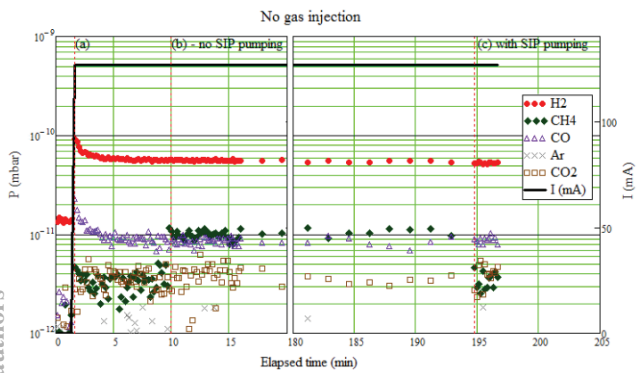


Figure 2: Gas dynamics without gas injection: (a) beginning of electron bombardment, (b) SIP closed, (c) SIP open.

### *Effect of External Pump without Gas Injection*

Figure 2 demonstrates the gas dynamics without gas injection: the pressure of all selected gas species has increased when a bombardment with 500-eV electrons started. After 10 min from beginning of the experiment the valve to SIP was closed ((b) in Fig. 2), the only partial pressure that increased (by factor 3) was $CH_4$, other species remain unchanged, no changes in all partial pressures were observed for the following three hours. Opening the valve to SIP ((c) in Fig. 2) caused a decrease of $CH_4$ pressure to a previous level while other species remain unchanged again.

### *Gas dynamics with $CH_4$ Injection*

Initially ((c) in Fig. 3 and Table 1), the electron bombardment continues without gas injection, at (d) the $CH_4$ injection with a flow $Q_1 = 5.0\times10^{-9}$ mbar·l/(s·cm$^2$) has started. The $CH_4$ partial pressure increased from $1.2\times10^{-11}$ to $3.4\times10^{-8}$ mbar. Two other species which also increased were $H_2$ (from $5.2\times10^{-11}$ to $3.7\times10^{-10}$ mbar) and CO (from $9\times10^{-12}$ to $2\times10^{-11}$ mbar), these species most likely appeared due to interaction of $CH_4$ with NEG, hot filament along the sample tube used for electron bombardment or filaments in the extractor gauge and RGAs.

At (e) the electron bombardment was stopped by applying zero bias to the filament without switching the filament off, i.e. the filament remains hot. The $CH_4$ pressure slightly increased to $3.7\times10^{-8}$ mbar while pressure of both $H_2$ and CO decreased: to $3.0\times10^{-10}$ mbar for $H_2$ and to $1.3\times10^{-11}$ mbar for CO. This effect could be explained by a superposition of two effects: electron stimulated desorption (ESD) of all species and electron stimulated cracking (ESC) of $CH_4$ molecules on the NEG surface bombarded with electrons, followed by additional $H_2$ desorption [14] and creating of CO molecules. Therefore, during electron bombardment the $H_2$ and CO pressures increase due to ESD and ESC, while $CH_4$ partial pressure increases due to ESD and decreases due to ESC and the latter is stronger in this case.

Table 1: Experimental Conditions

| Stage | SIP valve | $CH_4$ injection | I (mA) | Filament |
|---|---|---|---|---|
| Initial | open | 0 | 0 | ON |
| (a) | open | 0 | 126 | ON |
| (b) | closed | 0 | 126 | ON |
| (c) | open | 0 | 126 | ON |
| (d) | open | Q1 | 126 | ON |
| (e) | open | Q1 | 0 | ON |
| (f1) | closed | Q1 | 0 | ON |
| (f2) | closed | 0 | 0 | ON |
| (g) | closed | 0 | 126 | ON |
| (h) | closed | Q2 | 126 | ON |
| (i) | closed | Q2 | 0 | ON |
| (j) | closed | Q2 | 0 | OFF |
| (k) | closed | Q2 | 0 | ON |
| (l) | closed | Q2 | 126 | ON |

At (f1) the valve to SIP was closed, the pressure went up very fast and the injection was stopped after two minutes at (f2). After that the $CH_4$ pressure went down

without any external pump. This observation could only be explained by NEG coating pumping, contrary to

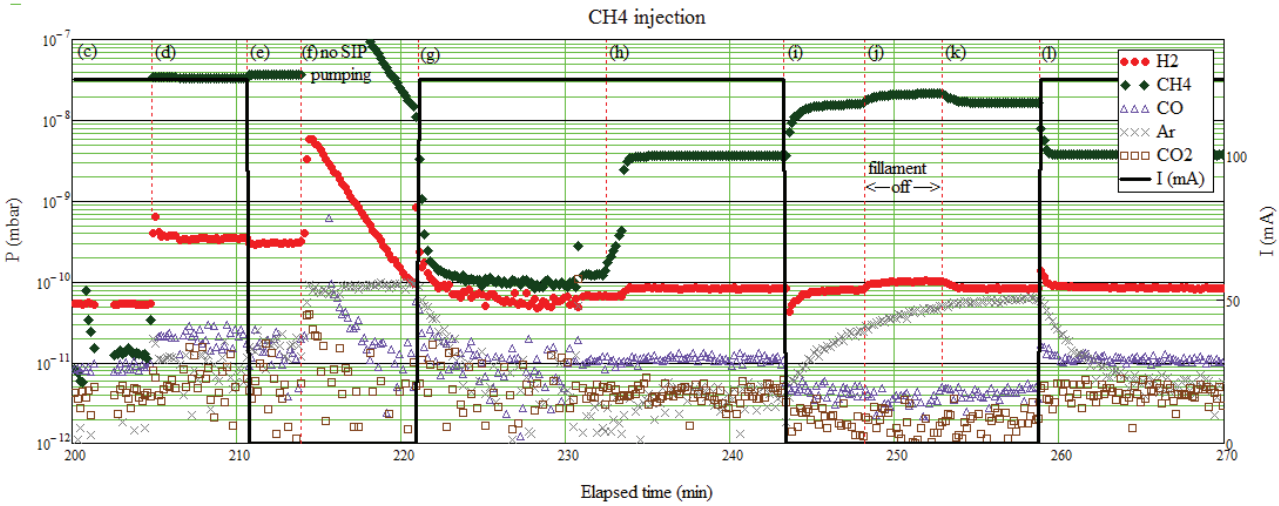


Figure 3: Pressure evolution during $CH_4$ injection.

common assumption that NEG could not pump $CH_4$. For comparison one can see that $H_2$, CO and $CO_2$ pressures are also reducing while Ar pressure stays constant.

At (g) the electron bombardment started again. The $CH_4$ pressure went rapidly down two orders of magnitude. $H_2$ indicated an initial increase due to ESC followed by a decrease due to reduction of $CH_4$ in the vacuum chamber. Ar pressure also decreased, indicating that although NEG coating does not usually pump Ar, it can pump Ar under the electron bombardment condition.

At (h) another $CH_4$ injection with a flow $Q_2 = 1.2\times10^{-9}$ mbar·l/(s·cm$^2$) has started. The $CH_4$ pressure increased from $1.0\times10^{-10}$ to $3.5\times10^{-9}$ mbar. With $Q_2 \ll Q_1$ only $H_2$ pressure increase was detectable (from $6\times10^{-11}$ to $8\times10^{-11}$ mbar). At (i) the electron bombardment was stopped with a zero bias without switching filament off. The $CH_4$ pressure increased to $1.5\times10^{-8}$ mbar while the pressure of both $H_2$ and CO decreased to $4\times10^{-10}$ and $2\times10^{-12}$ mbar respectively. Then at (j) the filament was switched off, the $CH_4$ pressure slightly increased to $2.1\times10^{-8}$ mbar, i.e. NEG coating pumping speed for $CH_4$ can be calculated as:

$$S_{NEG} = \frac{Q_2}{P} = 0.06\,\frac{l}{s\cdot cm^2} \qquad (1)$$

corresponding to a capture coefficient of

$$\alpha = \frac{S_{NEG}}{S_{ideal}} = 3\times10^{-3}. \qquad (2)$$

After switching the filament on at (k) the $CH_4$ pressure returns to the same level as before, showing that the hot filament pumping speed is $S_f \approx 0.02$ l/(s·cm$^2$). Switching on the electron bombardment at (l) brings all pressures to the values recorded before switching it off, i.e. electron induced pumping speed of NEG coating is $S_{NEG} \approx 0.27$ l/(s·cm$^2$) or $\alpha = 1.4\times10^{-2}$. Since pressure is not increasing during electron bombardment, all injected gas is pumped by NEG coating, then the electron stimulated pumping coefficient $\chi$ can be estimated as:

$$\chi = \frac{Q\left[CH_4/s\right]}{I\left[e^-/s\right]} = 2.3\times10^{-5}\left[\frac{CH_4}{e^-}\right]. \qquad (3)$$

## CONCLUSIONS

- Under electron bombardment the Ti-Zr-Hf-V NEG coating provides pumping of $CH_4$ by cracking it to $H_2$ and CO, as well as some pumping for Ar.
- Even without electron bombardment Ti-Zr-Hf-V NEG coating provides some pumping speed for $CH_4$.
- Since SR cause significant electron emission in accelerator vacuum chamber, it is very likely that similar effect of SR induced pumping of $CH_4$ and Ar can be observed.

## REFERENCES

[1] C. Benvenuti, "Non-evaporable getters: from pumping strips to thin film technology," EPAC'98, p. 200 (1998).
[2] O.B. Malyshev et al., Vacuum 100 (2014) 26.
[3] O.B. Malyshev et al., JVST A 27 (2009), 321.
[4] C. Benvenuti et al., JVST A 19 (2001) 2925.
[5] C. Benvenuti et al., Vacuum 71 (2003) 307.
[6] V.V. Anashin et al, Vacuum 75 (2004) 155.
[7] O.B. Malyshev et al., JVST A 28 (2010) 1215.
[8] O.B. Malyshev et al., Vacuum 86 (2012) 2035.
[9] K.M. Welch, *Capture Pumping Technology*, (Amsterdam: Elsevier, 2001), 240.
[10] B.T. Hogan and O.B. Malyshev, "Evaluation test of NEXTorr® D 100-5 pump". Cockcroft Institute Report - Cockcroft-13-47, STFC Daresbury Laboratory, Warrington, UK, October 2013.
[11] O.B. Malyshev et al., JVST A 27 (2009) 321.
[12] O.B. Malyshev et al., "Effect of surface polishing and vacuum firing on electron stimulated desorption from 316LN stainless steel". Accepted to publication by JVSTA.




ISBN 978-3-95450-132-8

[13] O.B. Malyshev et al., "Effect of surface polishing and vacuum firing on electron stimulated desorption from Ti-Zr-Hf-V coated 316LN stainless steel". Submitted to JVSTA.
[14] V.V. Anashin et al., Vacuum 60 (2001) 15.