THE EFFECT OF ACTIVATION DURATION ON THE PERFORMANCE OF NON-EVAPORABLE GETTER COATINGS

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Abstract

Non-evaporable getter (NEG) coatings can be activated at temperatures as low as 140°C. However, better pumping properties are achieved using higher temperatures, between 150-300 °C. This paper investigates whether using an increased activation *duration* can improve the NEG properties obtained using lower activation temperatures, and so decrease the energy and temperature requirement. This could allow a greater range of materials to be used in particle accelerator systems. Our findings have shown that increasing activation duration from 24 hrs to 1 week at 160 °C produces an improvement in the NEG pumping properties.

INTRODUCTION

With the aim of increasing the sustainability and efficiency of particle accelerators, non-evaporable getter (NEG) coatings have been increasingly used within their vacuum chambers, allowing tubes with larger aspect ratios (length over diameter) to be used, as their limited conductance is overcome by the distributed pumping provided by the NEG coating [1-5]. This reduces the need for external pumps, and decreases the required baking temperature to reach UHV, thereby decreasing the energy requirement of accelerators.

NEG coatings create a diffusion barrier in the inner walls of vacuum chamber for gas atoms, therefore reducing desorption from the walls. In addition, they absorb residual gas molecules desorbed from uncoated walls, resulting in lower pressures through which a beam can travel. NEG coating structure and composition can be varied to maximise certain properties, such as diffusivity and sorption capacity.

NEG-coated tubes must be activated following installation; a process done by heating to temperatures between 150 and 300 °C [2]. The activation process allows the gasses absorbed onto the surface of the NEG to either be desorbed and pumped away with an external pump (ex.: H_2, Ar, C_xH_y) or diffuse into its bulk (ex.: CO and CO_2), changing the surface state from oxide to metallic, allowing the NEG to absorb residual gases in the vacuum system. This activation process must be repeated in the case when NEG coated surface is saturated with CO, O_2 and CO_2.

Some level of activation has been shown to occur at levels as low as 140 °C [6]. However the lowest activation temperature depends on coating morphology, structure, composition, number of previous activations, etc.

In most previous studies, the NEG coating activation is performed for 24 hrs. However, it is well known for getter pumps that activation temperature can be reduced (within some limits) by increasing a duration of heating. As NEG coating utilises the same materials as these pumps, it is reasonable to expect similar effect take place. Therefore, with the effort to decrease the required activation temperature, this paper investigates the effect of a longer activation duration on NEG coating properties, to see if similar properties can be achieved as compared with 24 hrs at higher temperatures.

SAMPLE PREPARATION

The cylindrical magnetron deposition facility used at Daresbury Laboratory is described in detail in [7]. For each deposition, an alloy TiVZr wire target was used.

For this study, two samples were coated: a copper tube (S-Cu) with inner diameter ID = 20 mm, and an aluminium tube (S-Al) with ID = 36 mm. Both samples were 50 cm in length, and cleaned using a standard Daresbury Laboratory procedure [2] before deposition.

The deposition parameters used to coat each tube are summarised in Table 1. The pressure and power supply were varied with the aim to create different structures in the NEG coating. Pulsed DC was used to create dense coatings, which act as good barriers between the bulk wall material and the vacuum, and so a desirable to prevent degassing. Meanwhile, columnar coatings were created with DC power, along with an order of magnitude higher pressure. Columnar structures provide a larger surface area, and smaller grains, hence more grain boundaries which lead to better diffusivity and greater NEG capacity [6].

Deposition parameters were set with the aim to create a dense coating on S-Al, and a dual coating of dense and columnar layers on S-Cu.

Table 1: Deposition Parameters of NEG Coated Samples

Parameter	S-Cu	S-Al
Gas	Kr	Kr
Target	TiVZr	TiVZr
Layers	Dense/Columnar	Dense
Duration	2hrs 20m + 3 hrs 45m	5 hrs
Power Supply	Pulsed DC/DC	Pulsed DC
Pressure	$10^{-2} - 10^{-1}$ mbar	10^{-3} mbar
Power	70 W	70 W

The structure and composition of the coatings were characterised with scanning electron microscopy (SEM) and Energy Dispersive X-Ray Analysis (EDX).

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Table 2 shows the atomic percentages of the sample surfaces, as analysed by EDX. It can be seen on both samples that some of the tube material, beneath the coating (i.e. Cu and Al) appears under the analysis.

Table 2: Atomic Composition of NEG Coatings Determined by EDX

Element	S-Cu (at. %)	S-Al (at. %)
Cu	27.0	-
Al	-	28.3
Ti	22.5	21.7
V	29.5	33.4
Zr	21.0	16.6

Figures 1 and 2 display the SEM images of the surfaces of the samples. These images show the difference between the columnar (Fig.1) and dense (Fig.2) structures, with S-Cu displaying a rougher surface due to its columnar structured coating. This validates that the applied deposition parameters created the expected microstructure.
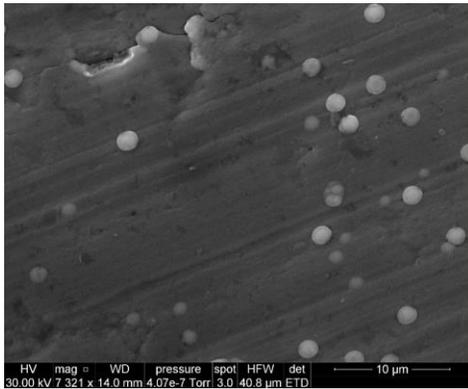
Figure 1: SEM image for S-Cu.
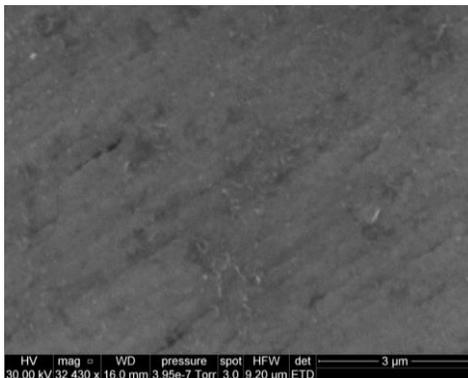
Figure 2: SEM image for S-Al.

EXPERIMENTAL PROCEDURE

Once deposited, the sample was installed on a facility from which the NEG performance could be assessed. This facility consists of a test chamber (TC) containing an extractor gauge (EG), and connected to a gas injection line and pumping line. Two residual gas analysers (RGAs) are in place on either end of the NEG-coated sample [8].

The facility allows the pumping properties of the NEG coating to be determined by gas injection into TC. During experiments, the partial pressures of the RGAs at either side of the tube were recorded along with the amount of injected gas.

After installation, the TC was pumped with a turbo-molecular pump (TMP), the facility was baked to 200 °C for 24 hrs, while the NEG coated sample was kept at 80 °C. The facility was then cooled to 150 °C and the RGAs and EG were switched on and degassed. The samples were heated to 140 °C for their first activation and all was held for two hours before the TC was returned to room temperature.

Following the first activation, the full bake-out procedure did not need to be repeated, and only the NEG-coated tube had to be heated for activation. Activation was run either for 24 hrs, as standard, or 1 week, to assess the effect of the longer duration.

After activation, the sample was left pumping overnight with a sputter ion pump (SIP). The following morning, the valve to SIP was closed, and the TC pressure recorded at $1 \times 10^{-10} - 1 \times 10^{-9}$ mbar, as measured by the EG in the TC. CO gas injection into the TC through a leak valve then commenced. The gas injection was run until the partial pressures measured by RGAs at either end of the NEG coated tube were equalised, indicating that the NEG coating was saturated.

Following injection, TC was then filled to a pressure of approximately 10^{-3} mbar and held for 0.5 hr to ensure full saturation of the NEG. The valve to the TMP was then opened, and the gas pumped away. The next activation process was then started.

For this experiment, 10 runs were performed, Runs 1-5 refer to S-Cu and Runs 6-10 to S-Al. For each sample, activation was performed at 140 and 180 °C for 24 hrs (Runs 1 and 2 for S-Cu, and Runs 6 and 7 for S-Al). To see the effect of activation duration, the samples were activated to 160 °C three times, varying the duration as 24, 168 and 24 hrs (Runs 3–5 for S-Cu and Runs 8–10 for S-Al, respectively).

RESULTS

The results are shown as the CO partial pressure ratio, R, plotted as a function of Q/A, where Q is the number of molecules injected into the system and A is the NEG coating surface area; see Figs. 3 and 4 for samples S-Cu and S-Al, respectively.

Two main results can be obtained from these graphs; firstly, sticking probability, α, defined as:

$$\alpha = 1 - \frac{Q_{out}}{Q_{in}}$$

where Q is the gas flow in and out of a surface. For an ideal pump, $\alpha = 1$. The sticking probability is quantified at the beginning of a gas injection from the partial pressure ratio, R_0, from the two RGAs:

$$R = (P_{bot} - P_{bot,0})/(P_{top} - P_{top,0}).$$

where $P_{i,0}$ is the partial pressures before gas injection began. The results of test particle Monte Carlo (TPMC) modelling performed earlier for each sample geometry [2, 9] are used to convert R_0 to α.

The second measured feature is sorption capacity, C, which is the amount of gas the NEG has absorbed and defined from the pressure ratio measurements when R is reduced to a defined value, R_c. In general, R_c is an artificial value, to compare similar samples. In this paper, $R_c = 10$ and $R_c = 2$ have been set, see results presented in Table 3.
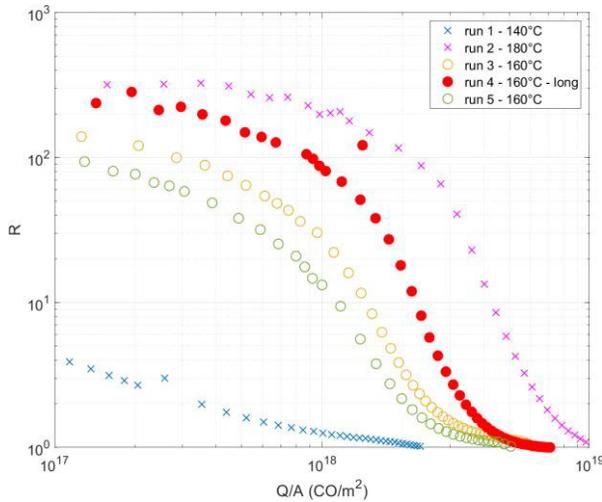
Figure 3: CO ratio as a function of injected CO for S-Cu.
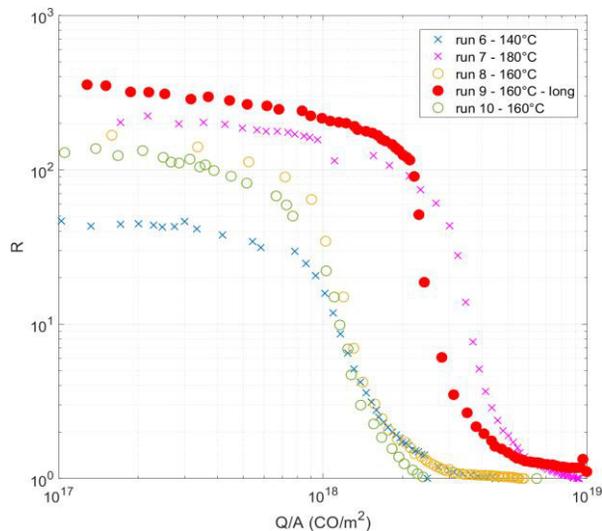
Figure 4: CO ratio as a function of injected CO for S-Al.

DISCUSSION

In Fig. 3, it is seen that $R_0 \approx 4$ for S-Cu activated at 140 °C, indicating $\alpha \approx 0.002$, which is too low for a substantial pumping effect from the NEG meaning sufficient activation has not occurred. However, activation is evident for Runs 2–10.

Despite undergoing the same activation profile, Runs 3 and 5 display slight difference in their pumping properties, with $\alpha = 0.06$ in Run 3 and $\alpha = 0.045$ in Run 5. Their sorption capacities at $R = 2.5$ are 2×10^{18} and 2.1×10^{18} CO/m². These differences could be a result of an aging effect on the NEG after multiple activations.

What is clearly seen, is the improvement of the initial sticking probability after Run 4 over Runs 3 and 5. After activation for 1 week, α now rivals that of Run 2. While it has a lower capacity, there is still great improvement over

that of Runs 3 and 5, demonstrating the positive effect of extended duration.

A similar effect is observed for S-Al in Fig. 4. Not only is an improved capacity shown between Run 9 and Runs 8 and 10, there is also a greater α for Run 9 at 160 °C than for Run 7 at 180 °C.

As before, both measurements taken after 24 hr activation at 160 °C, Runs 8 and 10 demonstrate similar trends, with very similar values for α. Additionally, their capacities at $R_c = 10$ and $R_c = 2$, along with those of Run 6 for 140 °C activation are sown to be approximately equal, with values around 1.2×10^{18} and 1.8×10^{18} CO/m², respectively.

Table 3: Pumping Properties of S-Cu and S-Al

Run	α	C at $R_c = 10$ (10^{18} CO/m²)	C at $R_c = 2$ (10^{18} CO/m²)
S-Cu			
1	0.002	N/A	0.3
2	0.15	4.3	6.7
3	0.06	1.5	2.5
4	0.1	2.2	3.4
5	0.045	1.1	2.1
S-Al			
6	0.03	1.1	1.6
7	0.1	3.6	4.8
8	0.08	1.3	1.8
9	0.3	2.6	4.0
10	0.07	1.2	1.8

These initial results from two samples demonstrate that, in a similar way to high temperature activation, a longer activation duration can lead to improved pumping properties. Further study is required to validate these initial findings, as described below.

CONCLUSIONS AND FUTURE PLANS

It has been demonstrated that increasing the duration of NEG activation at 160 °C can increase both sticking probability and sorption capacity, and can potentially reach the same pumping efficiency as a NEG coating activated to 180 °C for 24 hrs. Both S-Cu and S-Al have demonstrated an increase in CO capacity and sticking probability.

To continue this study, more NEG samples coated with various types of NEG should be produced to test if similar improvements can be obtained by extended durations at even lower temperatures, or in other words how much the activation temperature can be reduced and what duration is required. A similar study for different gasses (i.e. H_2 and CO_2) should also be provided in future.

Additionally, electron stimulated desorption (ESD) yield measurements should be taken on samples after different lengths of duration, to ensure these properties are also improved with longer temperature durations.

ACKNOWLEGDEMENTS

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