Deposition of Non-Evaporable Getter Thin Films and Vacuum Pumping Performances

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The ERL (Energy Recovery Linac) proposed at Cornell University requires extreme high vacuum (XHV) conditions in many of the sub-systems, such as the photo-cathode electron gun, beam pipes adjacent to the superconducting cryo-modules, and the insertion devices [1]. Non-Evaporable Getter (NEG) thin films deposited onto the interior wall of the vacuum components is a very promising path to XHV. This project involved deposition of NEG thin films and vacuum pumping performance tests of the NEG films.

I. INTRODUCTION

Extreme high vacuum conditions will be required for many sub-systems, particularly the photo-cathode electron source, in the proposed ERL, in order to obtain and maintain high quantum efficiency [2]. To achieve and maintain the extreme high vacuum conditions in a limited space allowable, new challenges need to be met by the vacuum design team.

The use of NEG (Non-Evaporable Getter) thin film deposited onto the components used in a vacuum system has revolutionized the design of vacuum systems. The NEG film brings pumping to sources of gas-loads; it provides distributed pumping in a space-limited environment and has a very low outgassing rate [1].

Vacuum Performance of NEG films deposited using argon as sputtering gas has been studied previously. This project entailed the comparison of the performance of the Titanium-Zirconium-Vanadium (TiZrV) NEG thin films, deposited using krypton (Kr) and using argon (Ar) as the discharge gas, onto the interior wall of stainless steel pipes. Krypton is being used as a discharge gas, even though it's a more expensive gas, because some tests have indicated that argon embeds itself in the NEG film. The embedded argon may reduce the quality of the NEG film. The aim of this project was to compare the NEG film pumping performance between krypton and argon as the discharge gas.

II. NEG THIN FILM DEPOSITION

NEG thin films can be deposited onto the objects that go inside a vacuum, using many different techniques such as Triode Discharge Devices, Magnetrons, RF Sputtering, Ion-beam sputtering, Planar Diode and the DC Glow Discharge [3]. The TiZrV NEG films were deposited using the DC Magnetron Sputtering technique. The deposition system is shown in Figure 1. The DC Magnetron Sputtering technique was employed due to its simplicity and lower sputtering gas pressure. The SST pipe to be coated was pumped by an 80 l/s turbo-molecular pump (TMP) through a gate valve, which was used to control the pumping speed of the coating chamber. A solenoid was placed around the SST pipe to generate the magnetic field. A sputtering cathode, formed by twisting 1 mm diameter wires of Zr/Ti/V, was connected to a SHV feedthrough at the top and a ceramic standoff at the bottom. Krypton was released into the system using a variable leak valve; and the Kr pressure in the sputtering chamber was controlled by the combination of the gate valve and the leak valve. Electron cloud was formed around the negatively biased cathode from the cross field (the DC electric field and the magnetic field) arrangement. Kr was ionized by the electron impact in the electron cloud and the Kr^+ ions were accelerated towards the cathode. Due to energized particles (ions) colliding with the cathode, atoms of TiZrV were freed from the cathode and traveled to the material to be coated to form thin films. The sputtering parameters, listed in Table I, were optimized to provide maximum allowable sputtering current at minimum krypton pressure.

Table I – Sputtering parameters

Magnetic Field	Cathode Voltage	Kr Pressure	Sputtering Current
230 Gauss	600 V	60 mtorr	~ 25 mA

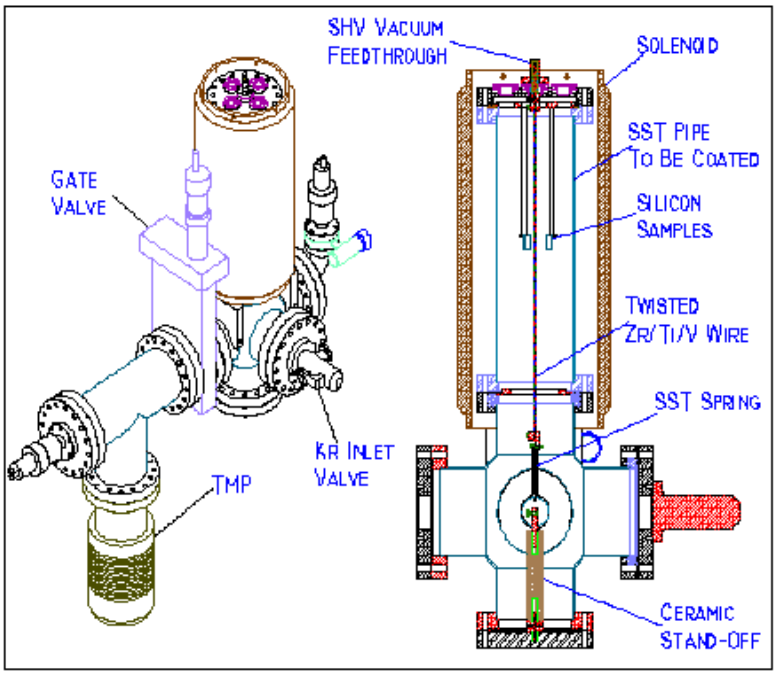

Figure 1 – NEG Film Deposition System

The deposition rate of the film can be calculated using:

$$R_{growth} = \frac{I_{ion} \cdot Y_{Sputter}}{2\pi aL \cdot q_e \cdot n_{NEG}} (nm/\sec) \qquad (1)$$

where I_{ion} is the sputtering ion current in *mA*, $Y_{sputter}$ is the average sputtering yield, a is the radius of the SST pipe in *cm*, L is the effective length of the sputtering plasma in *cm*, q_e is the elementary charge of an electron in *Coulombs*, and n_{NEG} is the atomic density of NEG film in cm^{-3}. $Y_{sputter}$ and n_{NEG} can be calculated using equation 2 and 3, respectively.

$$Y_{sputter} = 1.92 - 2.02e^{-.781E} \qquad (2)$$

$$n_{NEG} = \frac{1}{x+y+1}(xn_{Ti} + yn_V + n_{zr}) \qquad (3)$$

The Kr sputtering yield was compiled from Journal of Applied Physics, and Nuclear Instruments and Methods; and can be fitted by the empirical formula (2), where E is the Kr^+ energy in keV. The n_{Ti}, n_V, n_{Zr} in equation 3 are the atomic number densities of pure Ti, V, Zr metals listed in Table II. The x and y are the fractional atomic ratios of Ti and V to Zr, respectively, determined by

Rutherford Back Scattering (RBS) measurement. The average values of *x* and the *y* were 1.05 and 1.85, respectively. The *a* and the *L* for the SST tube were 4.92 cm and 36.35 cm, respectively.

The recorded film growth rate and accumulated film thickness are shown in Figure 2 and Figure 3, respectively, as a function of deposition time. The average film deposition rate was 14.1 nm/sec and the final film thickness was 1.7 μm.

Table II – Atomic number densities [4]

	Titanium	Zirconium	Vanadium
$n_{(cm^{-3})}$	$5.62*10^{22}$	$4.2*10^{22}$	$6.82*10^{22}$

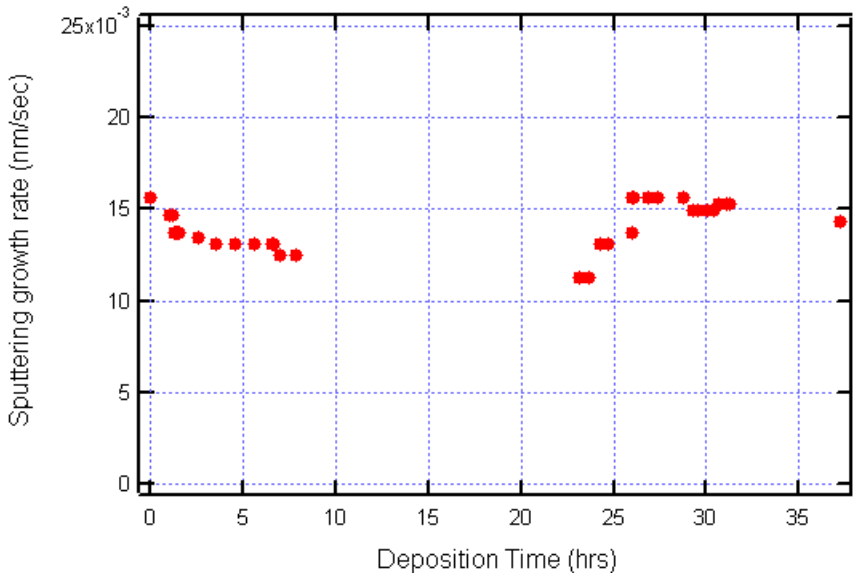

Figure 2 – NEG thin film growth rate

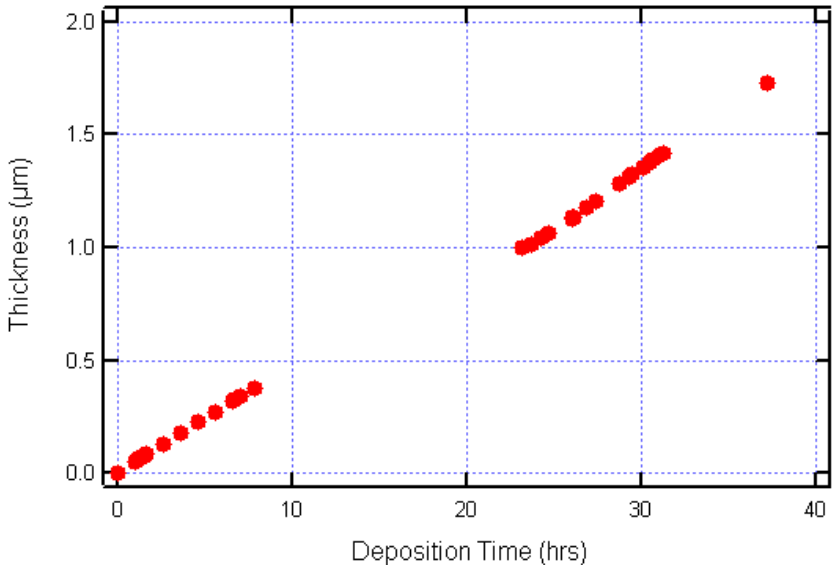

Figure 3 – Accumulated NEG film thickness vs. deposition time

III. NEG FILM PUMPING PERFORMANCE

Once the SST pipes were coated with TiZrV, they were installed in a testing apparatus shown in Figure 4, to evaluate its vacuum performance. A test gas, usually CO or N_2 was introduced from a calibrated leak at the top, the gas flows through the coated pipe, a 0.25" diameter orifice, and then pumped by a TMP at the bottom. Inverted magnetron cold cathode gauges are used to measure the pressures above (P_1) and below (P_2) the orifice [1]. After the initial pump down, the whole system is baked in order to reduce any outgassing from other components in the testing apparatus. After cooling down from a 150˚C / 24 hours bakeout, the base pressure was about 1.0 x 10^{-9} torr.

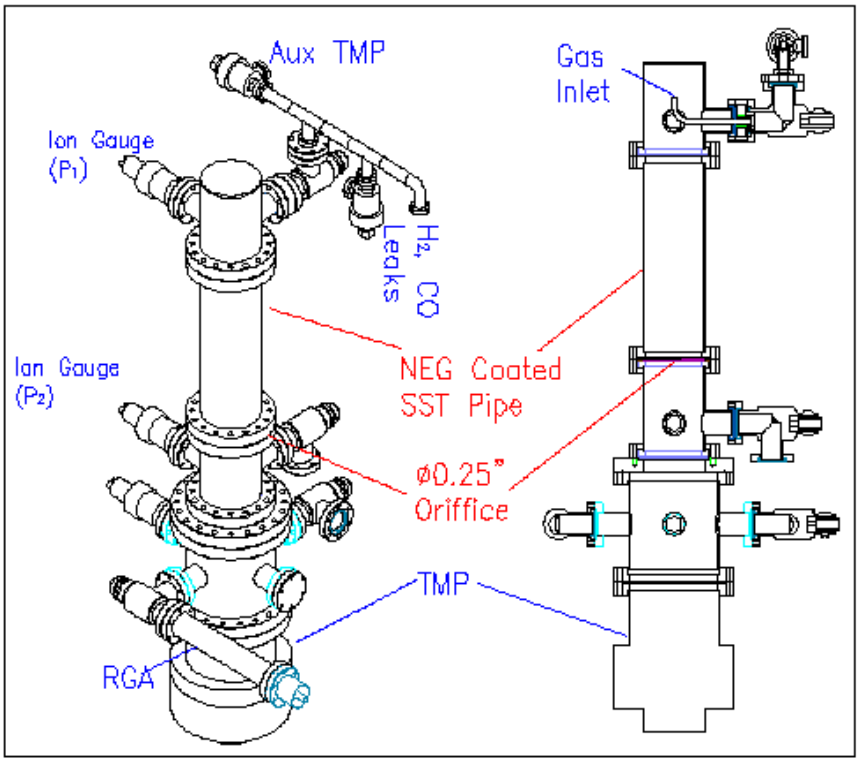

Figure 4 – NEG film vacuum pumping test apparatus

The SST tube with NEG coating was then activated at various temperatures ($t_{act} \geq 150$˚C) for 48 hours. All the pumping tests are done after the SST tube cooled down close to ambient temperature. The pumping performance of the NEG films was tested by a test gas. The test gasses were introduced using a crimped capillary leaks to ensure reproducibility of the gas leak rate for each test. The leak rate for N_2 and CO were measured and are listed in Table III. The test gases were introduced into the system and the pressure P_1 and P_2 were recorded until the NEG film was saturated. The pumping speed of the NEG film can be calculated from the measured P_1 and P_2:

$$S_{NEG} = \frac{\dot{Q}_{leak} - C(P_1 - P_2)}{P_1} \qquad (4)$$

where $\dot{Q}_{leak}$ is the calibrated leak of the test gas (listed in Table III), and C is the conductance of the orifice. The amount of gas the NEG film pumped, Q_{pumped}, can be calculated using:

$$Q_{pumped}^{CO,N_2} = \int (S_{NEG}^{CO,N_2} \cdot P_1)dt \qquad (5)$$

Table III – Leak rate for CO and N_2

Gas	Leak Rate (torr • l/s)
Carbon monoxide	3.23×10^{-5}
Nitrogen	6.04×10^{-6}

IV. PUMPING TEST RESULTS

After activating the NEG coating at different activating temperatures ranging from 150˚C to 350˚C for 48 hours, the NEG coating was saturated by leaking carbon monoxide into the SST tube. During the test, the pressures (P_1 and P_2) were recorded. The calculated pumping speed was plotted against amount pumped and the results are displayed in Figure 5. The NEG film pumping performance may be represented by the initial pumping speed and the pumping capacity of each activation cycle. The initial pumping speed, $S_{initial}$, is defined as the measured pumping speed in the beginning of the saturation, which measures the NEG film reactivity after the activation. The pumping capacity, $Q_{capacity}$, is defined as amount of pumped gas to reduce the pumping speed to a small level when the NEG film needs to be re-activated.

Based on the data shown in Figure 5, the $S_{initial}$ and the $Q_{capacity}$ are plotted as a function of activation temperatures in Figure 6 and Figure 7, respectively.

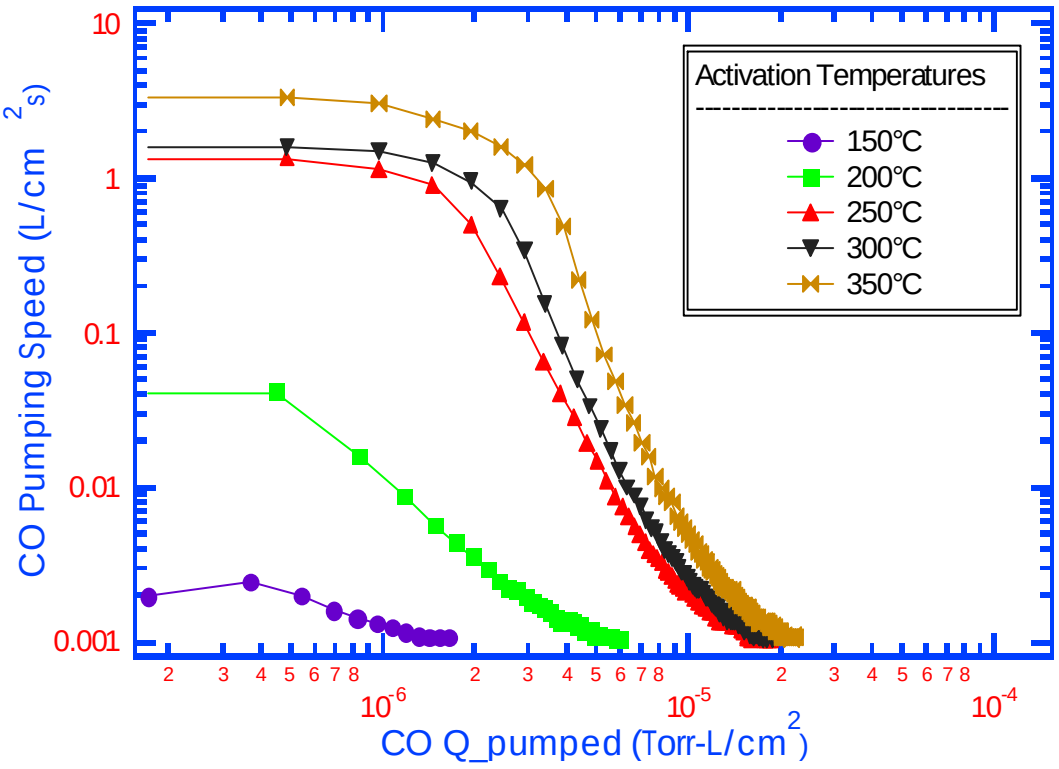

Figure 5 - Pumping Speed vs. Q_{pumped}

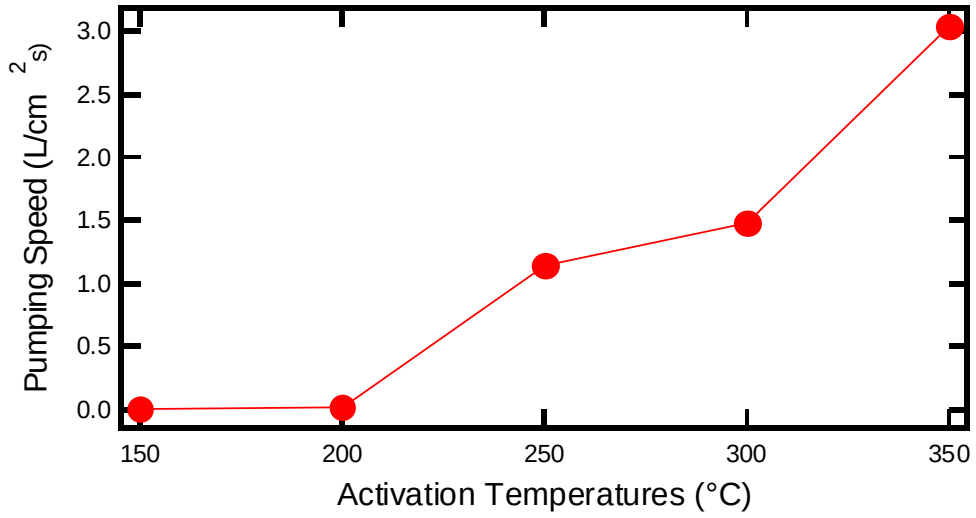

Figure 6 – Initial Pumping Speed vs. Activation Temperatures

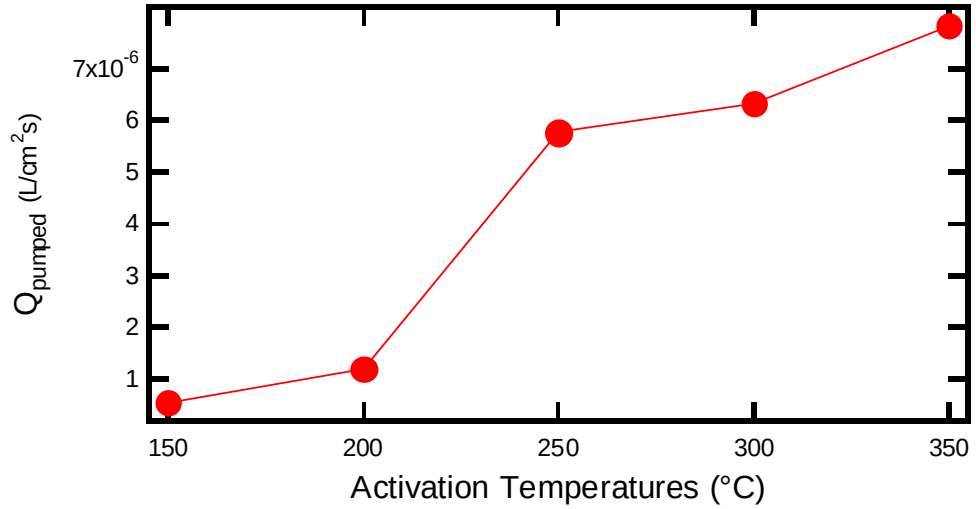

Figure 7 – Pumping Capacity vs. Activation Temperatures

V. DISSCUSSION

The pumping tests for NEG films deposited using argon as the discharge gas were performed previously and are shown in Figure 8, 9, and 10. As one can see the pumping speed for NEG films deposited using krypton, is much greater than the pumping speed for NEG films deposited using argon. This increase in the pumping speed could correspond to the fact that the NEG film deposited using krypton has a rougher surface texture, thus improving the adsorption rate. The pumping capacity for NEG films deposited using krypton is comparable to the NEG films deposited using argon. As for the activation temperature, the NEG film deposited using argon as the discharge gas seems to provide better pumping performance at $T_{act} < 200°C$, than the NEG film deposited by krypton sputtering.

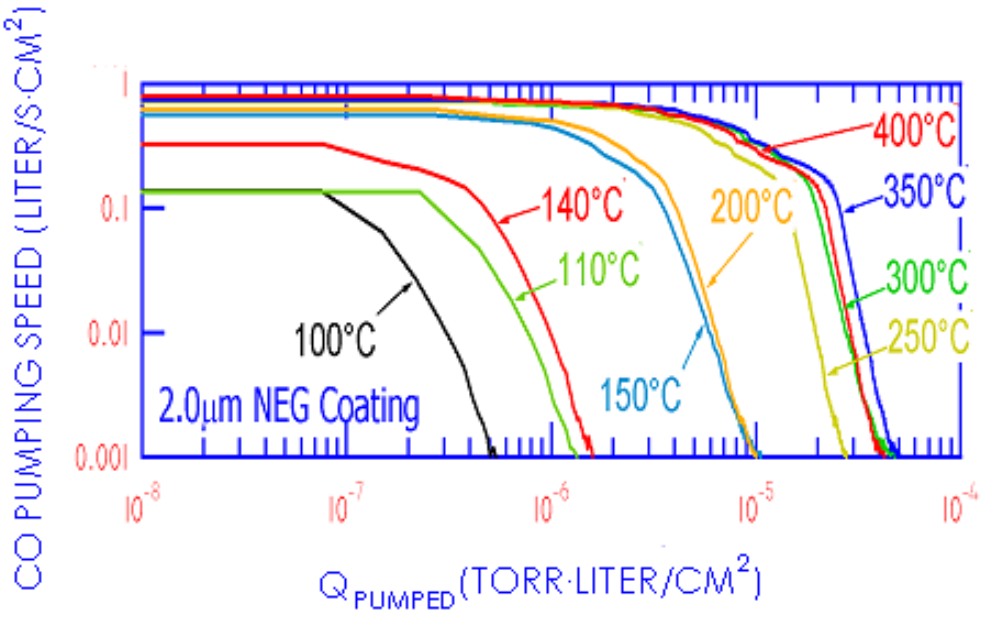

Figure 8 – Pumping Speed vs. Q_{pumped} of Ar-Sputtered NEG film

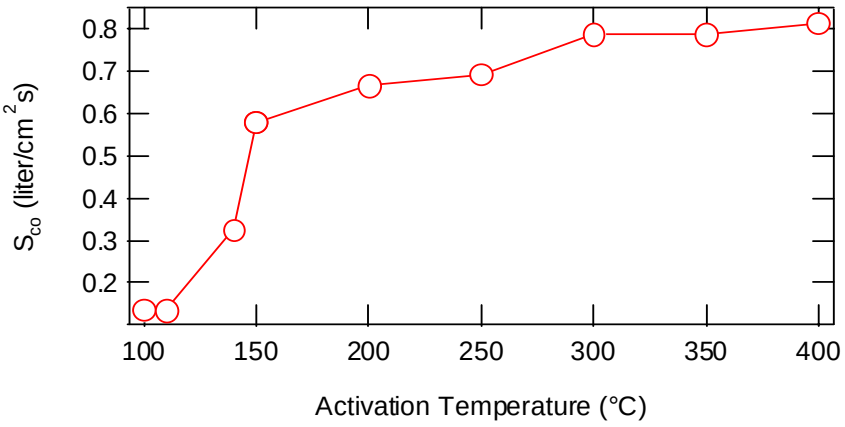

Figure 9 – Initial Pumping Speed vs. Activation Temperatures of Ar-Sputtered NEG film

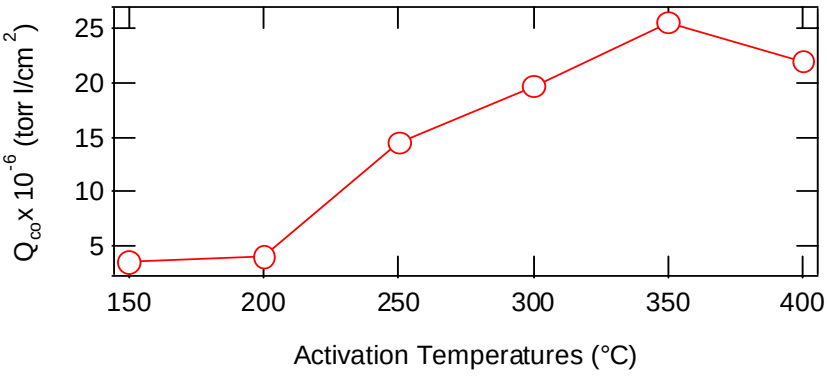

Figure 10 – Pumping Capacity vs. Activation Temperatures of Ar-Sputtered NEG film

VI. CONCLUSION

The NEG thin film was deposited using DC magnetron Sputtering onto SST pipe, using krypton as the discharge gas. It seems to have outperformed the NEG film when argon was used as the discharge gas. After studying the factors that affect the vacuum pumping performance, activation above 250°C, of the NEG thin films deposited using krypton as the discharge gas significantly improves its performance in a vacuum. This was a preliminary study, more studies are planned to confirm these results.

VII. ACKNOWLEDGMENTS

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VIII. REFERENCES

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