Deposition of Non-Evaporable Getter Films and Hydrogen Pumping Measurements

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Abstract

Thin film getters are used as pumps in many ultra high vacuum (UHV) and extreme high vacuum (XHV) systems. A 0.6 micron thick Ti/V/Zr was deposited using a DC Magnetron Sputtering technique. We measured an intial pumping speed as high as 0.40 liters/sec cm^2 and capacities in the range of 10^{-2} torr liters / cm^2 of H_2 for 350°C activation temperature. The results showed that it would be practical to implement NEG pumping in the vacuum chamber containing the ERL gun.

Introduction

The Non-evaporable getter (NEG) is set of element that adsorbs gas in one of two ways. The first way of adsorbing gas being through the process of chemisorption on the surface of the material. The second method of pumping is through the process of a gas dissolving into the bulk of the material.

Thin film NEGs have many desirable characteristics that make them indispensible for attaining an extreme high vacuum. The thin film can be placed in close proximity to outgassing sources. It has an additional addvantage of covering the the surface of the stanless steel pipes, as the NEG film has low amount of outgassing.

The Energy Recovery Linac (ERL) is a linear accelerator that is proposed to be built in the future at Cornell. Its primary purpose is a to be a source of high energy x-rays for research in a broad range of fields including biology, crystalography, and soft condensed matter. The various parts of the accelerator include a DC photoemission gun and many superconducting radio frequency cavitities (SRF cavitities).

Inside the vacuum chamber, near the photoemission gun the pressure must be kept at extreme high vacuum conditions, with pressure less than 10^{-12} torr, or residual gas ion backscattering will occur, damaging the GaAs cathode of the gun. In order to acheive this pressure a NEG would be ideal due to its capacity to be distributed across the entire interior of the gun chamber.

Though the cold surfaces fo the SRF cavities adsorbs H_2 and other gases, they have a very limited capacity for hydrogen.

Many NEGs have high capacity and pumping speed for H_2. TiZr based NEGs have been found to have low activitation temperatures around 300 °C, in addition to high pumping speeds [1]. For these low activation temperatures and other properties that we decided to try Ti/V/Zr.

Experiment

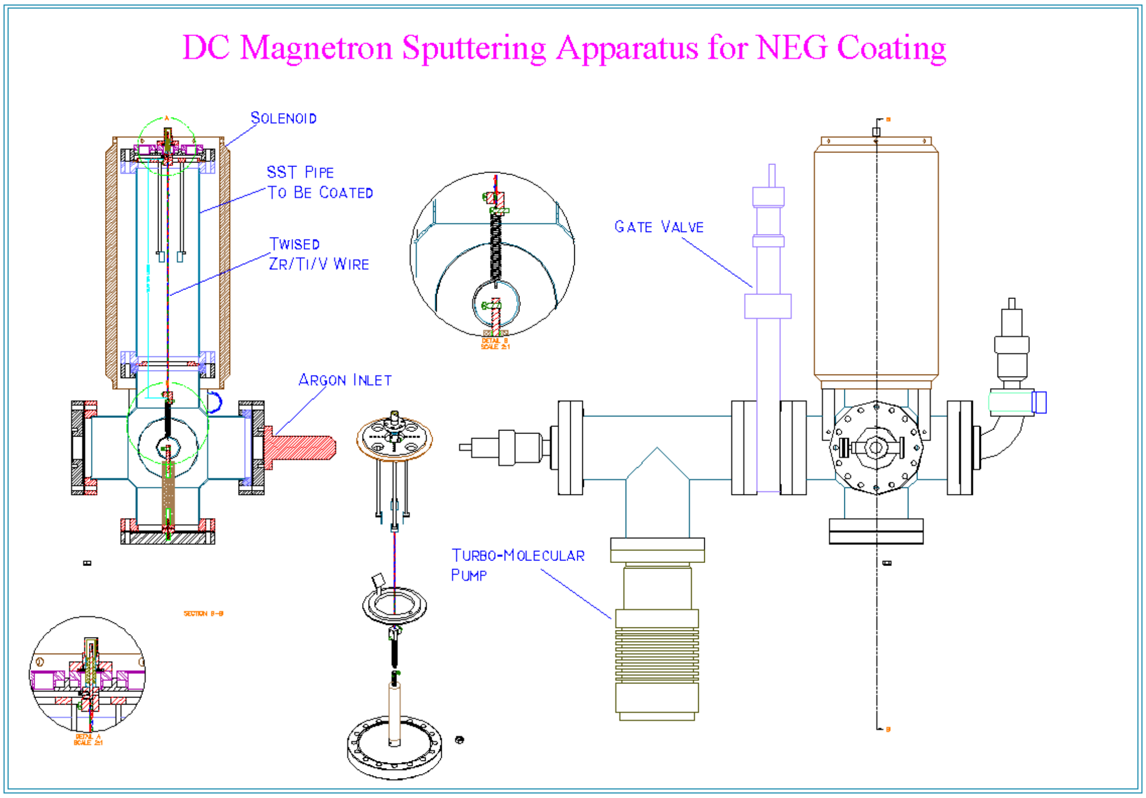

FIGURE 1. The apparatus used to coat our pipes with the NEG film.

We use the DC magnetron sputtering technique to deposit the NEG film on the surface of a Stanless Steel (SST)test pipe. A voltage is placed across the filament and a magnetic field is applied using the solenoid. The resulting cross-field reduces the amount of argon pressure needed to induce sputtering. The lower argon pressure causes a smaller number of collisions between sputtered filamentment elements and argon ions resulting in a better adhesiveness. The sputtering gas, Argon, is let in throught the argon inlet, a variable leak vavle. Flow of argon is maintained by the turbomolecular pump, and controlled by a combination of variable leak valve and the gate valve. The filament wire is made by twisting 1mm diameter wires of Titinium, Vanandum, and Zirconium and it is attached to a spring at the bottom of the vacuum chamber to allow for thermal expansion shown in Fig. 1.

During the intial weeks we spent some time checking the purity of the argon gas using a Residual Gas Analyzer (RGA). The RGA measurement indicated that our initial argon gas contained an large amount of impurities, which resulted in a NEG film that does not pump.

Before coating the pipe, we clean the chamber by sputtering the pipe *in situ* with argon by reversing the polarity of the applied voltage. Ionized argon atoms are accelerated towards the walls of the pipe to be coated and collide with it. In these collisions the argon ions dislodge some gas molecules that have adsorbed on the surface of the pipe which are then pumped away by the turbo pump.

The sputtering process is performed by first pumping the pressure down to around 10^-8 torr and turning on the solenoid. We found that it helped to turn on the solenoid hours earilier to allow the temperature to stablize. Then we opened the variable leak valve to increase argon flow and closed the gate valve to rescrict flow out of the chamber. Once argon is flowing into the chamber we turned on the voltage to the filment and began sputtering. Typically the system would fluctuate a little and we need to watch ion current and voltage to keep them low enough so as not to burn up the filament.

The formula used to determine the rate of deposition is as follows:

$$R_{growth} = 4.70 \frac{I_{ion} Y_{sputter}}{a} (nm/sec) \tag{1}$$

where a is the radius of the target pipe, I_{ion} is the argon ion current, and $Y_{sputter}$ is the sputtering yield. The ion current is measured throughout the sputtering and sputting yield was found by performing a fit to data aquired in another experiment[2]. This equation was confirmed by Rutherford Backscattering Spectrometry.

Typical parameters for sputter were 600 Volts on the cathode, 20 mA of argon ion current, and 10^{-2} torr argon pressure. With these the typical deposition rate was approximatly 50 nm / hour.

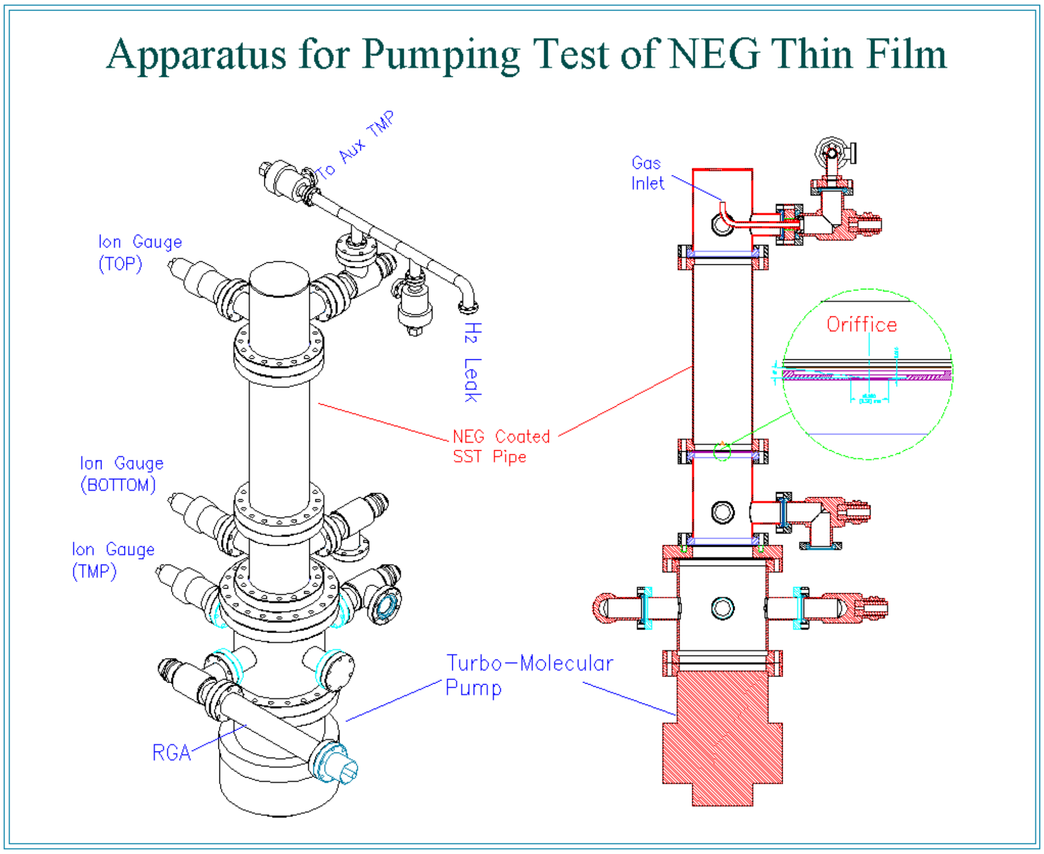

FIGURE 2. The apparatus used to make pumping speed measurements of the NEG film.

The method of measuring the pumping speed of the NEG film that we used is a modified version of the AVS standard flow method for measuring pumping speed. The flow of gas is inwards through a gas inlet directed towards the top of the chamber to randmize the the path of gas molecules flowing into the chamber. An orifice with a diameter of 1/4 inch is used to define flow out of the NEG chamber. There are three main pressure gauges on the vacuum system which are connected to a computer to take readings at regular intervals. These gauges are the cold cathode gauges (CCGs) above and below the orifice and near the turbomolecular pump which all shown in Fig. 2.

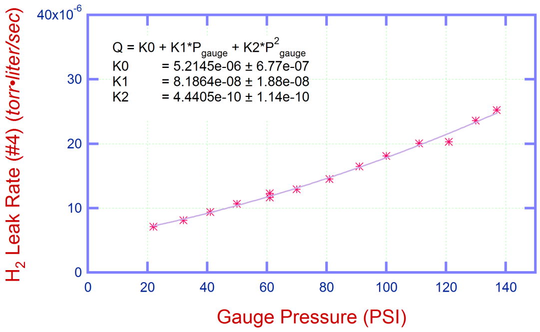

FIGURE 3. Plot showing the Leak Rate with increasing Hydrogen Pressure.

The flow rate into the chamber was measured prior to performing the pumping speed measurements. The formula for calculating the flow rate is

$$Q_{leak} = C(P_{top} - P_{bottom}) \tag{2}$$

where C is the conductance across the orifice, P_{top} is the pressure in the top chamber, P_{bottom} is the pressure in the bottom chamber, and Q_{leak} is the flow into the chamber. The leak rate calibration measurements are done with a saturated SST pipe, so the net pumping of the pipe is negligible. During that time the gauges were also calibrated by comparing the voltage readout to the displayed pressure on the CCGs. The plot of leak rate versus increasing H_2 pressure is shown in Fig. 3.

The pumping speed is calculated by

$$S_{NEG} = \frac{Q_{leak} - C(P_{top} - P_{bottom})}{P_{top}} \tag{3}$$

where C is the conductance across the orifice, P_{top} is the pressure in the top chamber, P_{bottom} is the pressure in the bottom chamber, and Q_{leak} is the flow into the chamber which is measured eariler.

During the activation of the pipe hydrogen is desorbed and is pumped through the orifice into the turbo and the Residual Gas Analyzer. Using the information from the RGA we were able to calculate the amount of hydrogan gas desorbed which matches closely with the amount hydrogren adsorbed in later tests. Desorption was calculated simply by

$$Q_{H_2desorbed} = \int S_{turbo} P_{H_2} \mathrm{d}t \tag{4}$$

where S_{turbo} is the speed of the turbomolecular pump and P_{H_2} is hydrogen pressure in the RGA.

Results

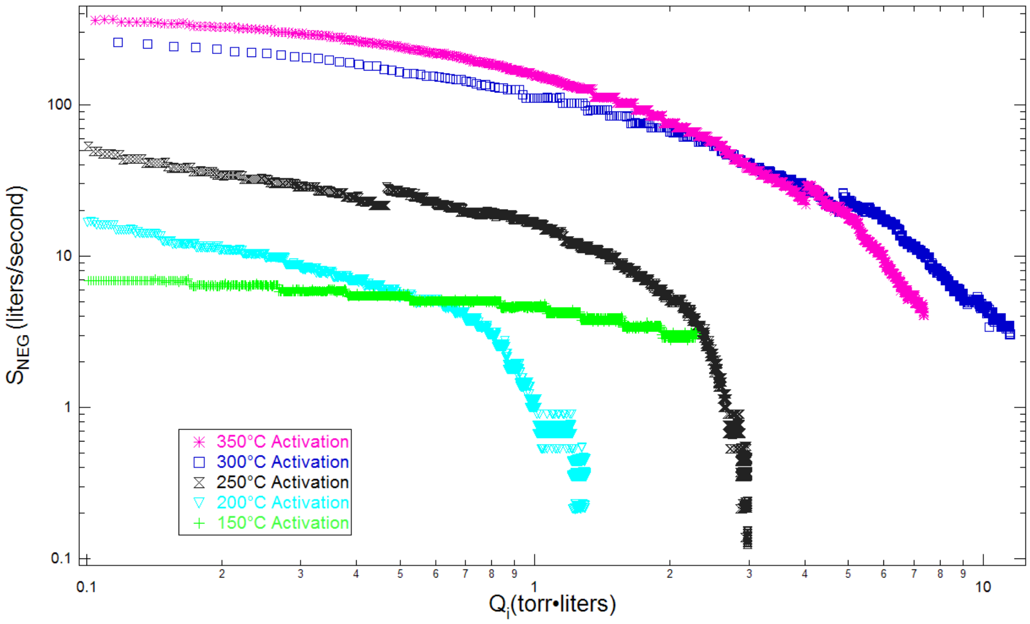

FIGURE 4. The pumping speed versus H_2 adsorbed for a given activation temperature, for a 24 activation duration.

The intial pumping speed shows a general trend of increasing pumping speed with increasing activation temperature in Fig. 4. As the NEG absorbs more hydrogren the pumping speed decreases.

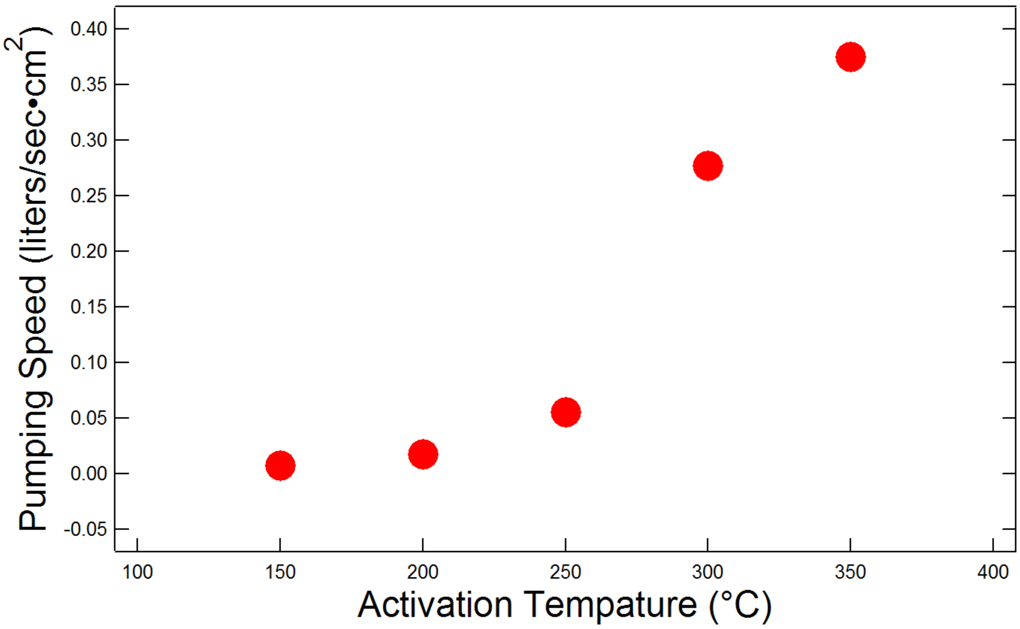

FIGURE 5. Intial pumping speed is show for various activation temperatures, for a 24 hour activation duration

To show NEG surface reactivity, the inital pumping speeds are shown in Fig. 5 with a trend of increasing pumping speeds with increasing activation temperatures. From Fig. 5 you can see a threshold activation temperatue at approximately 250°C.

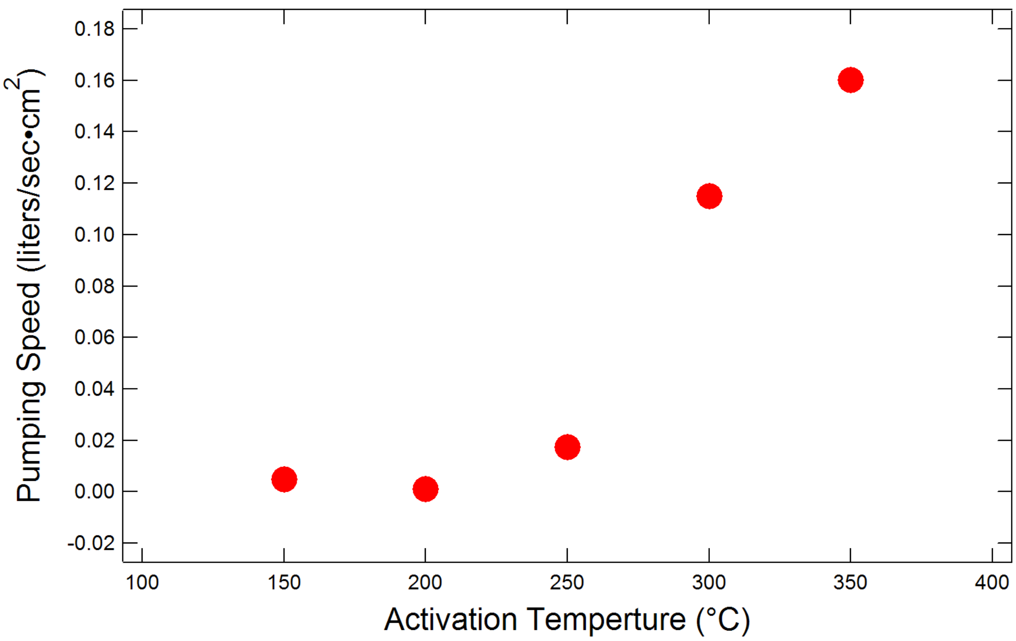

FIGURE 6. The pumping speed versus 24 hour activation temperature

The pumping speed after 10^{-3} torr liters / cm^2 is shown in Fig. 6. This plot is one way of representing H_2 capacity. Again, there is a trend of increasing pumping speed with increasing activation temperature and a threshold activation temperature at approximately 250°C in Fig. 6.

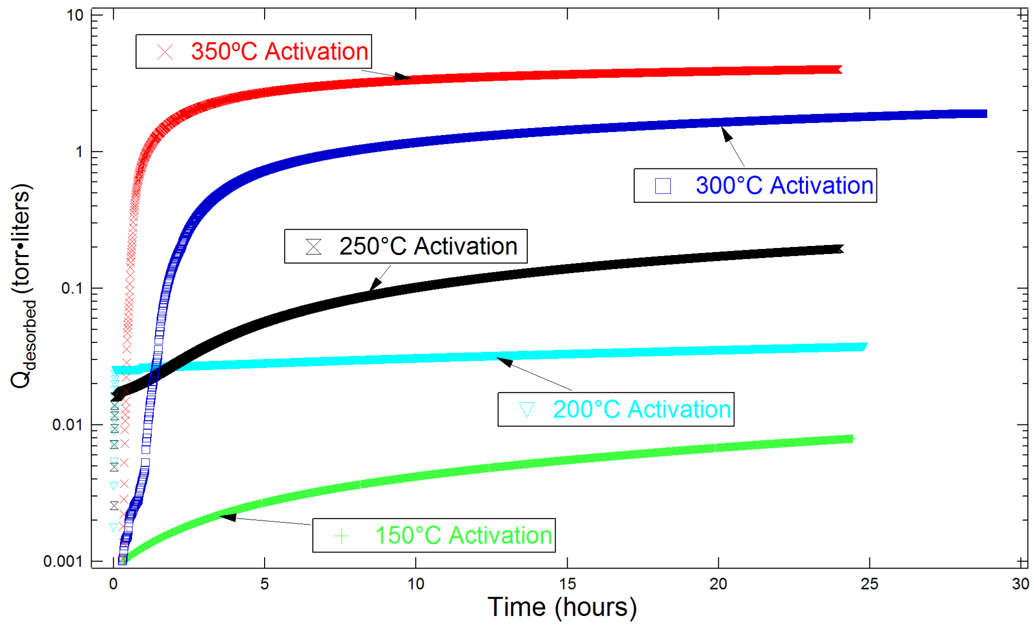

FIGURE 7. The amount of gas adsorbed during activation

The amount of gas desorbed is shown time in Fig. 7 and a trend of increased desorbtion with higher temperature is shown. The total amount of gas desorbed in Fig. 8 is comparable

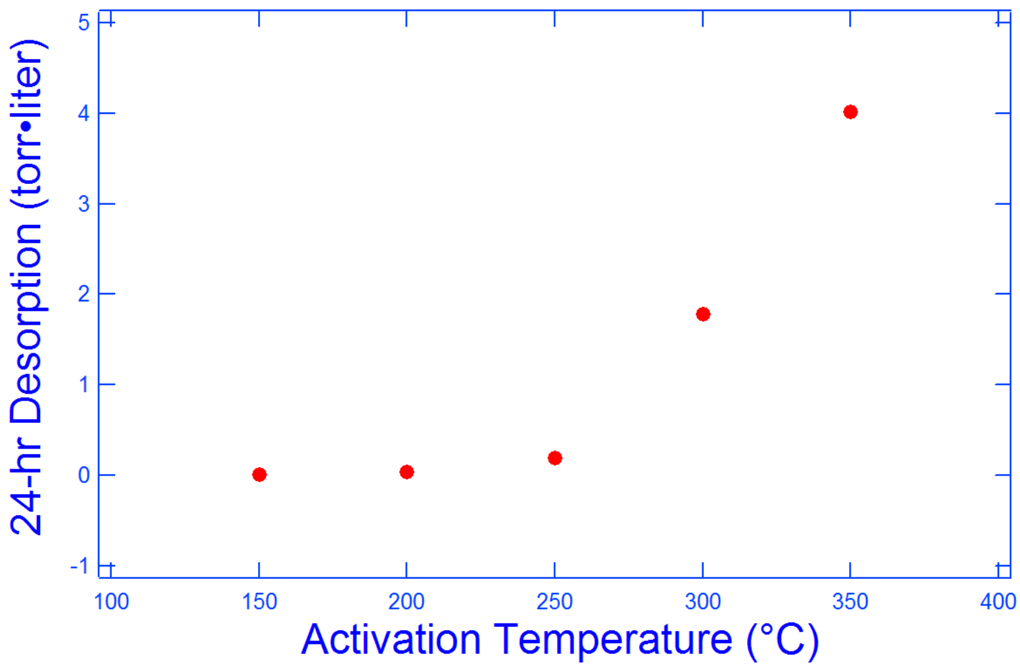

FIGURE 8. This a plot total amount of H_2 desorbed for a 24 hour activation temperature

with the about of gas adsorbed in Fig. 4. The amount of gas desorbed over a 24 hour period increases with temperature and again there is a threshold at approximately 250°C.

Conclusions

A 0.6 μm thick Ti/V/Zr NEG is deposity using DC Magetron Sputtering. The results show that Ti/V/Zr NEG films have significant pumping speed and capacity for H_2. The pumping speed and the capacity of the NEG increases with activation temperature. Finally, the NEG appears to have a threshold activation temperature at 250°C which indicates that the NEG practical for use around heat sensitive systems.

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Footnotes and References

1. Benvenuti C. et. al. Vacuum. **53**, 219 (1999).
2. Insler Jonathan. Sputtering Yield of Ti/Zr/V. http://cesrlog.lns.cornell.edu/mslog/vacuum/r_d/neg_coating/yieldcalcs.html .