Application of NEG Coatings on a Metal Surface and Measurements of its RF Properties

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The Energy Recovery Linac (ERL) at Cornell University will require the use of thin film coatings for purposes of achieving Ultra High Vacuum (UHV). At this point, various pipe coatings have been widely used in the field of particle accelerator physics. The vacuum properties of pipe coatings are well documented and have given rise to its popularity in the field. A Non-Evaporable Getter (NEG) coating is one such coating. NEG coatings responses to beam induced RF are at this point not well understood. This paper will address the development of research techniques at Cornell University designed to understand the RF properties of NEG coatings.

I. INTRODUCTION

The development of UHV in particle accelerators ensured a sufficiently long beam lifetime [1]. The main issue in achieving UHV for particle accelerators is due to surface outgassing from the all-metal vacuum system [2]. Surface bombardment from electrons, ions, and photons releases gas in the vacuum chamber, thus negatively affecting the performance of the beam. To make matters even more challenging, it is often the fact that the space available for conventional pumping is very limited. Therefore, accelerator vacuum systems are usually gas conductance limited. This led to the development of thin-film coatings as a countermeasure against both thermal and bombardment induced outgassing.

CERN began studying the possibilities of NEG coatings in UHV and many positive characteristics were quickly noticed. NEG coatings act as an *in situ* getter pump, trapping chemically reactive molecules to the surface of the chamber at a high pumping speed. Once the molecules are trapped and the surface is saturated, the NEG can be activated, typically at temperatures from 200-400° C, to diffuse the molecules into the bulk of the coating. This leads to an extremely clean surface capable of reducing desorption due to photons and electrons [3]. Furthermore, the physical barrier the NEG creates works to decrease the effects of thermal outgassing. Together, NEG coatings have been seen to provide valuable vacuum pumping and reduction of outgassing.

The reduction of the Secondary Electron Yield (SEY) of a surface is highly important in particle accelerators when positively charged beam (such as positrons, protons, ions, etc) performance may be hampered due to resonant electron multiplication [4]. Buildups of electron clouds have the adverse effect of influencing each incoming bunch. A low SEY works to reduce the growth rate of an electron cloud, and thus protects performance of the beam. Studies have shown that NEG coatings significantly reduce the SEY from a value of 2 for a stainless steel surface to 1.1 for an activated NEG coating

surface [5]. The NEG coating was also found to reduce the electron desorption rate up to a factor of 100 as compared to a stainless steel surface. Saturation of the NEG coating changes the SEY to a value of 1.2, still a very low value compared to a bare surface.

Particle accelerators contain long conductance limited vacuum chambers where more traditional pumps, such as a sublimation pump, would be inefficient [2]. The NEG coating is used to create local distributive pumping throughout the vacuum chamber by coating the surface of the entire chamber. The NEG coating is deposited on the surface *ex situ* to ensure all the surfaces are coated. Once the chamber is assembled the NEG coating can be activated by *in situ* baking.

At Cornell, the ERL project requires UHV for tight spaces in the undulators used for x-ray production. The local pumping abilities and *in situ* activation properties make the NEG particularly attractive for use in such small spaces [6]. Due to the very short bunches to be used in the ERL beam, concern about the NEG coatings response to RF has become an issue, especially due to the beams close proximity to the surface. The goal is to understand the NEG's impedance at high frequency bands to determine whether there will be any negative effect on the beams performance.

II. COATING PROCEDURE

DC Magnetron Sputtering is the preferred method of coating due to its simplicity and preservation of stoichiometry. Sputtering is ideal for uniform coatings of long, narrow vacuum chambers [2]. DC Magnetron Sputtering has a fast deposition rate for relatively low pressure of sputtering gas and has also shown good adhesiveness.

The coating system used for this study is shown in Figure 1. An equiatomic cathode of Ti, Zr, and V is chosen. The cathode is formed by twisting together 1mm diameter wires of each element. The alloy of TiZrV has been shown to be a very promising as a NEG film and has a low activation temperature of 180-250° C [7] [8]. The wire is attached to a power supply via SHV type electric vacuum feedthrough, and the opposite end is spring attached to a ceramic standoff at the bottom of the apparatus to keep the cathode at tension during coating.

The Sputtering Process begins with the chamber pumped down using an 80 *l/s* turbo molecular pump (TMP). A good vacuum pressure of around 10^{-9} torr is maintained until process of sputtering. Ultra high purity Ar gas is introduced into the chamber through the use of a Varian leak valve. The argon pressure during sputter is ~5 mtorr, as measured by a capacitance gauge. At this point, a magnetic field of 100-150 Gauss is introduced to the chamber via a solenoid coil surrounding the sputtering chamber. A negative potential is applied to the cathode typically between 600-800 V. The negative bias of the cathode produces ionized Ar gas through the process of field ionization. The electrons now in the sputtering chamber would be biased to move away from the negatively charged cathode wire, but the magnetic field is tuned to capture these electrons so that they circulate around the cathode and increase the rate of ionization due to electron impact ionization. The Ar ions eject atoms from the cathode target via energetic ion bombardment. During sputtering a steady discharge current of 20-25 mA is carefully maintained.

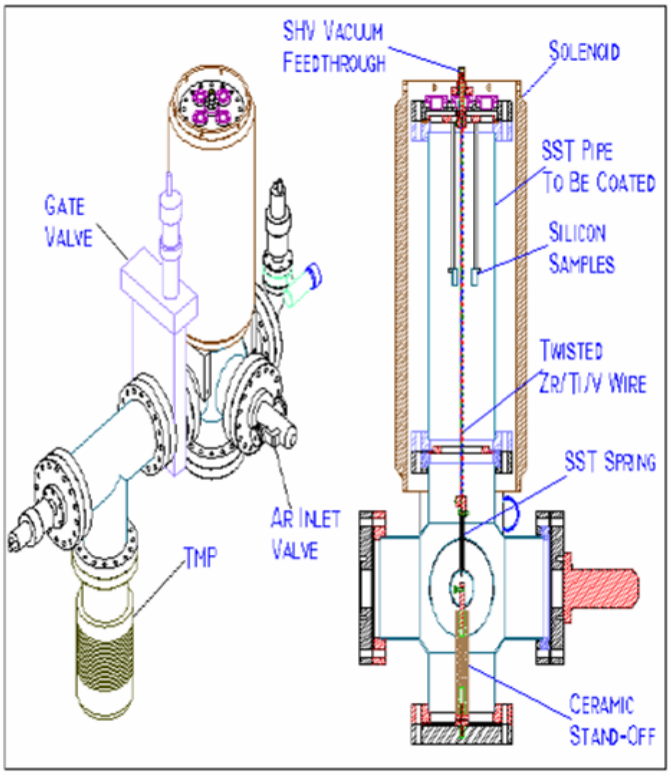

Figure 1. Sputtering Chamber Apparatus

A coating rate can be expressed as follows:

$$R_{atom} = \frac{N_{atom}}{A} = \frac{N_{ion} \cdot Y_{sputter}}{A} = \frac{I_{ion} \cdot Y_{sputter}}{2\pi aL \cdot q_e} \tag{1}$$

where N_{atom} is the number of Ti, Zr, or V atoms sputtered per second, N_{ion} is the amount of Ar ions bombarding the cathode per second, $Y_{sputter}$ is the average sputtering yield of Ti/Zr/V and approximately given by the equation $Y_{sputter} = 1.61 - 1.65e^{-.957E}$ *(E in keV)*, I_{ion} is the discharge current measure in Amps, q_e is the charge of the electron in Coulumbs, and $A=2\pi aL$ is the inner area of the of the pipe or sample to be coated.

The rate of growth of the coating is expressed as:

$$R_{growth} = \frac{R_{atom}}{n_{NEG}} \tag{2}$$

where n_{NEG} is the atomic number density of the NEG coating and is determined from Rutherford Backscattering Spectrometry. Finally, plugging in known values of $L=36.35cm$, $q_e=1.602*10^{-19}$ *C*, $Y_{sputter}=0.75$ *at 682 eV*, and $n_{NEG}=5.83*10^{22}$ *atoms/cm*^{3} *we* have:

$$R_{growth} = 3.517 \cdot \frac{I_{ion}}{a} nm/\sec \tag{3}$$

where *a* is the distance between the cathode and the coating surface.

III. MEASURING RF PROPERTIES

The most feasible way to measure a NEG coatings response to RF was through the use of a Vector Network Analyzer (VNA). The VNA measures transmission and reflection of electrical signals at a range of frequencies. A VNA is able to measure both amplitude and phase properties of the electrical signal making it possible to calculate the permeability μ and the permittivity ε of a sample. In our experiment, the sample was originally intended to be a ceramic tile placed inside a waveguide with one side containing the NEG coating. This technique was too difficult to provide meaningful results due to complications including reflection from the ceramic tile itself disrupting the reflection signal from the NEG coating alone. Under these circumstances it becomes mathematically difficult to calculate where power losses in the transmission and reflection waves are coming from. In the end, the sensitivity of our equipment was unable to perform this experiment.

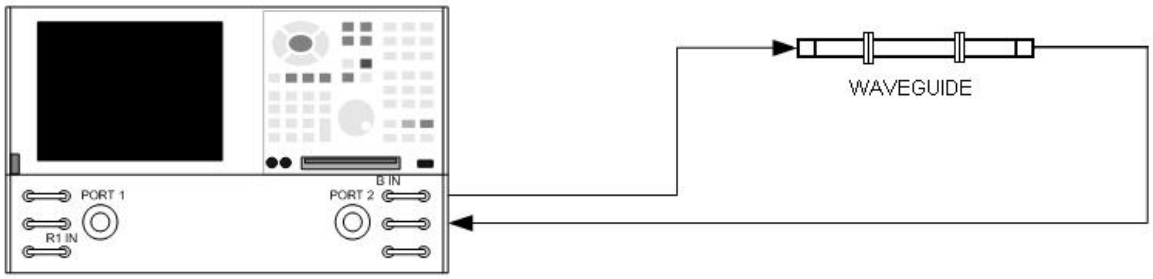

Figure 2. Diagram of the S21 signal from the VNA being sent through a waveguide.

In order to create a fair experiment in which the transmission and reflection waves would be dependent on the NEG alone a new waveguide design was adopted. A waveguide, shown in Figure 3, needed to be coated with the NEG so that we could test the RF with the VNA. Due to the geometry of waveguides, it was not possible to sputter evenly on the entire inner surface of the waveguide at one time. Instead, one of the four inner walls of the waveguide was coated with the NEG. A waveguide design including a U-channel and a plate was developed. The plate was mounted in the sputtering chamber and coated accordingly. The coated plate is then attached to the U-channel thus completing the waveguide. The U-channel is also attached to two flanges through the use of ream and bore holes to ensure alignment. A TRL-calibration (Thru, Reflect, Line) of the VNA was done before measurements. TRL-calibration is the most accurate calibration used for precise measurements. Assembly of the waveguide and VNA system was done using torque wrenches to guarantee consistency of contact forces.

It is possible to calculate the percentage of total power losses from the broad and narrow sides of the waveguide using the equations:

$$P_b = \int_0^{\Lambda/2}\int_0^{a} \frac{H_x^2 + H_z^2}{\sigma \cdot \delta} \cdot dxdz \tag{4}$$

$$P_n = \int_0^{\Lambda/2}\int_0^{b} \frac{H_z'^2}{\sigma \cdot \delta} \cdot dydz \tag{5}$$

where $H_x = H_0 \frac{2a}{\Lambda} \sin\left(\frac{\pi \cdot x}{a}\right) \cos\left(\frac{2\pi \cdot z}{\Lambda}\right)$, $H_z = H_0 \cos\left(\frac{\pi \cdot x}{a}\right) \sin\left(\frac{2\pi \cdot z}{\Lambda}\right)$, and $H_z^{'} = H_0 \sin\left(\frac{2\pi \cdot z}{\Lambda}\right)$ for a TE_{10} wave. Power is dependent on the square of the magnetic field inside the waveguide. A ratio of losses in the broad wall, or coated wall, to total losses can now be found for our frequency range of 12.4 - 18 GHz. The ratio r is calculated by the equation:

$$r = \frac{P_b}{2(P_n + P_b)} \tag{6}$$

and found to be 0.324 for 12.4 GHz and 0.361 for 18 GHz.

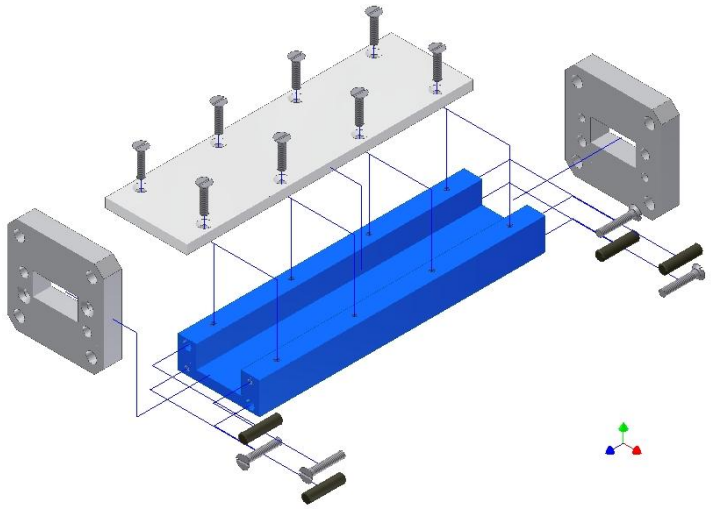

Figure 3. Assembly Diagram of Waveguide designed and used in this study.

IV. RESULTS

Plates underwent polishing and ultrasound cleaning before they were mounted in the sputtering chamber at a distance of 3.744 cm from the TiZrV cathode. A stable discharge current of 25 mA was maintained during sputtering. Using Eq. (3) we find that $R_{growth} = 0.023485 nm/\sec$. Two plates were coated with a NEG coating of 1 μm corresponding to 12 hours of sputtering, and the other a NEG coating of 2 μm corresponding to 24 hours of sputtering.

Following calibration of the VNA, an uncoated waveguide was tested for baseline measurements. Comparison between these measurements and those from the coated waveguides can be seen below.

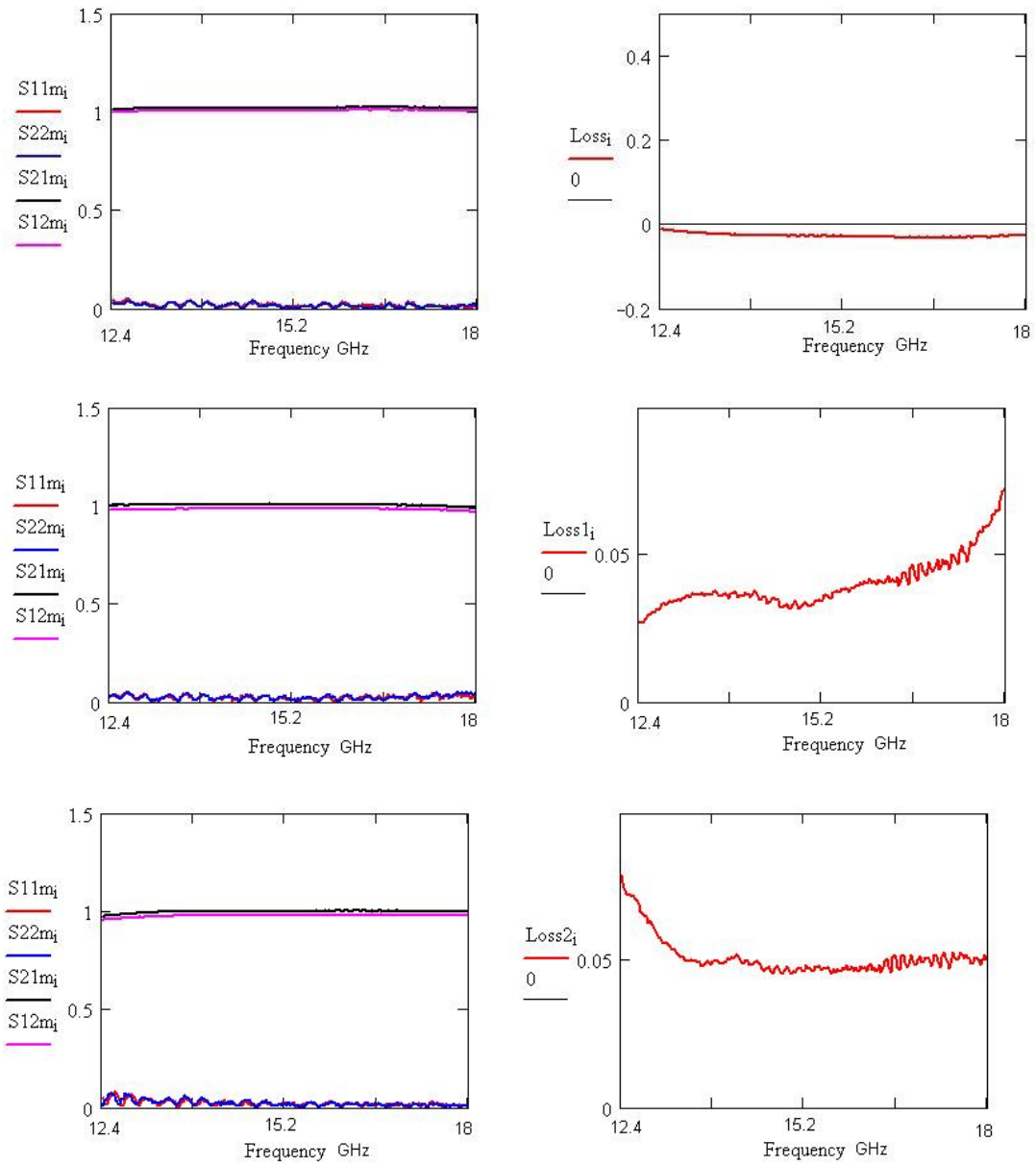

Figure 4. VNA measurements of uncoated waveguide (1st row), 1 μm NEG coating (2nd row), and 2 μm coating (3rd row), respectively. The y-axis represents the amplitude of each signal. The S11 signal refers to the reflection signal from port 1 of the VNA, S22 is the reflection signal from port 2, S12 is the transmission signal from port 2 going to port 1, and S21 is the transmission signal from port 1 to port 2.

Losses on the right hand pictures are calculated as:

$$Loss = 1 - |S21|^2 - |S11|^2 \tag{7}$$

The measurements clearly have a substantial amount of error associated with them as can be seen in the negative power loss recorded for the uncoated waveguide. This error was taken into account, so that in measurements with coated plates the originally

measured data for the uncoated plate was subtracted. The coated waveguides had a power loss of ~5% for both coating thicknesses.

V. ANALYSIS AND CONCLUSIONS

To better understand how the NEG coating could increase the amount of power lost in the waveguide we studied the effects of the coating thickness compared to the skin layer and also the conductance of the coating compared to that of the metal. For the metal the current density $j = j_0 e^{-\Delta/\delta_c} e^{-(z-\Delta)/\delta_m} \cdot \frac{\sigma_m}{\sigma_c}$ can be integrated along with the current density of the surface coating $j = j_0 e^{-z/\delta_c}$ to find the total current I which can be used to solve for $j_0 = \frac{I}{\delta_c \left[1 + \left(\frac{\delta_c}{\delta_m} - 1\right) e^{-\Delta/\delta_c}\right]}$ when Δ, the coating thickness, is small. When a ratio between the power losses of the coating and metal, P_{c+m}, and the power losses of the metal, P_m, is made we have:

$$\frac{P_{c+m}}{P_m} = \frac{\sigma_m \delta_m}{\sigma_c \delta_c} \cdot \frac{\left[1 + \left(\frac{\delta_c}{\delta_m} - 1\right) e^{-2\Delta/\delta_c}\right]}{\left[1 + \left(\frac{\delta_c}{\delta_m} - 1\right) e^{-\Delta/\delta_c}\right]^2} \tag{8}$$

A graphical representation of Eq. 6 is shown below:

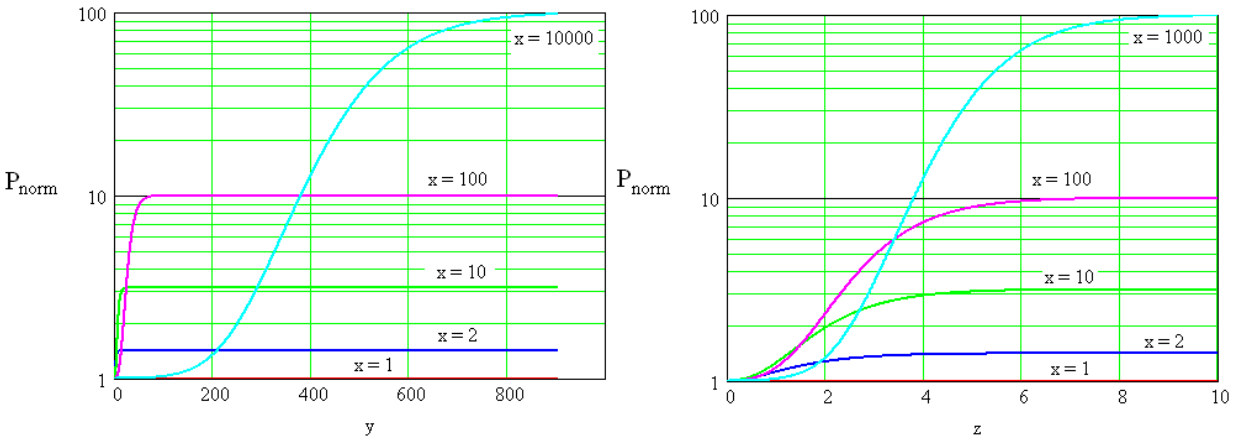

Figure 5. $x = \frac{\sigma_m}{\sigma_c}$ is the ratio of conductivities between the metal and coating.

$y = \frac{\Delta}{\delta_m}$ is the ratio of the coating thickness to the skin layer of the metal.

$z = \frac{\Delta}{\delta_c}$ is a ratio between the coating thickness and the skin layer of the coating.

P_{norm} is the ratio of losses between a coated metal surface and an all-metal surface.

The attenuation α of an Aluminum waveguide was calculated to have a value α=0.372 dB/m at f = 12.4 GHz and α=0.273 dB/m at f = 18 GHz. The length L of our waveguide was 0.114 m. Within our frequency range, the amplitude of the signal S21 would be between 0.995-0.996 according to this attenuation. In order to see significant power losses, such as S21~0.7, would require a conductivity σ in the coating material to be 10^4 times less than the conductivity for Aluminum. This scenario is represented in Figure 5 as the light blue line *x=10,000*. The peak of this line corresponds to a coating thickness more than 800 times that of the skin layer of metal which is approximately equal to 0.7 μm. Therefore, a coating thickness of over 500 μm would be required to see the desired power losses. Under the current technique of sputtering, a coating of this thickness would be impractical to complete or use in UHV.

The impedance of the NEG coating is difficult to study at the thickness typically used in experiment. As far as can be seen, the operational thickness of the NEG coating insufficiently absorbs enough RF to be used for these purposes alone. However, the NEG's primary purpose of acting as a distributed pump capable of suppressing electron cloud growth can still be taken advantage of as it seems the coating does not affect the beam itself due to insignificant changes in impedance.

Ferrite materials are possibly better suited for the task of absorbing large amounts of RF power (like they are used in the Higher Order Mode Loads in the vicinity of the RF cavity) and should be explored in more detail if power absorption remains a concern.

VI. ACKNOWLEDGEMENTS

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[1] P. Chiggiato, R. Kersevan. "Synchrotron radiation-induced desorption from a NEG-coated vacuum chamber", Vacuum 67-72 (2001).

[2] C. Benvenuti, P. Chiggiato, F. Cicoira, Y. L'Aminot. "Nonevaporable getter films for ultrahigh vacuum applications", *American Vacuum Society* (1998).

[3] Chiggiato P, Cazeneuve JM, Johanek V. CERN-EST-SM-DA/98-03 note.

[4] B. Henrist, N. Hilleret, C. Scheuerlein. “The Secondary Electron Yield of the Non Evaporable Getter Alloys TiZr and TiZrV”, *Vacuum Technical Note* 98-08 (1998).

[5] S. Y. Zhang, H. C. Hseuh, T. Roser. “NEG Coating at RHIC”, C-A/AP/#99, June 2003.

[6] Yulin Li, D Hess, R. Kersevan, N. Mistry. “Design and pumping characteristics of a compact titanium-vanadium non-evaporable getter pump”, Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films -- May 1998 -- Volume 16, Issue 3, pp. 1139-1144.

[7] “A novel route to extreme vacua: the Nonevaporable getter film coatings”, C.Benvenuti, et al., Vacuum 53 (1999) p.219-225.

[8] C. Benvenuti, P. Chiggiato, A. Mongelluzzo, A. Prodromides, V. Ruzinov, C. Scheuerlein, and M. Taborelli. “Influence of the elemental composition and crystal structure on the vacuum properties of Ti–Zr–V nonevaporable getter films”, Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films -- November 2001 -- Volume 19, Issue 6, pp. 2925-2930 (2001).