VACUUM PUMPING STUDY OF TITANIUM-ZIRCONIUM-VANADIUM THIN FILMS*

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Abstract[*]

Vacuum pumping via non-evaporable getter (NEG) thin film deposited directly onto the interior of a vacuum chamber is a novel way to achieve extreme high vacuum. As part of R&D efforts for the proposed Energy Recovery Linac at Cornell, the pumping performance of Titanium-Zirconium-Vanadium (TiZrV) NEG thin films was investigated to provide 'engineering' data for designing vacuum systems using such NEG thin films. The compositions and growth rates of the NEG thin films, deposited on stainless steel tubes using DC Magnetron sputtering, were investigated using Rutherfield Back-scattering Spectrometry. The pumping speeds and capacities of the thin films for CO and H_2 were measured as functions of activation temperatures and durations, and film thickness. Though pumping of CO and H_2 by the NEG films is observed with activation temperatures as low as 150°C, the pumping performance of the NEG films improves significantly with activation temperatures above 250°C.

INTRODUCTION

An Energy Recovery Linac (ERL) prototype machine was proposed at Cornell University to study ERL-related accelerator physics and technology [1]. The knowledge and experience gained from the prototype ERL machine will pave the way for the development of a full scale ERL-based new generation synchrotron light sources.

Adequate vacuum performance is essential to the operation of many sub-systems of the proposed ERL prototype and the future ERL synchrotron light sources (such as the photo-cathode injector, beam pipes adjacent to the superconduting cryo-modules, and the insertion devices). The challenge to the vacuum system design arises from the need to achieve and maintain ultra-high and extreme-high vacuum conditions within a very limited space allowable for traditional pumping.

The use of non-evaporable getter (NEG) thin film [2] directly deposited onto the inside of a vacuum chamber has revolutionized the design of accelerator 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 and a low secondary electron emission yield [3]. The NEG thin films have been employed in many accelerator systems [4] with very successful vacuum performances.

As part of the R&D efforts for the ERL project, we studied the pumping performance of the Titanium-Zirconium-Vanadium (TiZrV) NEG thin films, deposited on the interior of stainless steel (SST) pipes. The TiZrV

coating has the attractive feature [2] of a low activation temperature. One of the aims of the study is to provide vacuum 'engineering' data for designing vacuum systems using such NEG thin films.

NEG THIN FILM DEPOSITION

The TiZrV NEG films are deposited onto the interior wall of SST tubes, using a DC Magnetron Sputtering technique. The deposition system is shown in Figure 1. The SST tube (with an outer diameter of 4-in, welded between two 6-inch Con-Flat flanges) to be coated is mounted onto a 6-inch six-way cross. The deposition system is pumped by an 80 l/s turbo-molecular pump (TMP). The sputtering cathode, formed by twisting together 1-mm-diameter titanium, zirconium and vanadium wires, is connected to an SHV-type vacuum feedthrough at the top, and to a ceramic standoff at the bottom via a SST spring. UHP argon gas is introduced into the sputtering system through an adjustable leak valve. The Ar flow rate and pressure in the sputtering chamber are adjusted using the leak valve and a gate valve located between the sputtering chamber and the TMP. The Argon discharge is ignited and sustained by a DC voltage applied to the cathode and a magnetic field generated by a coaxial solenoid coil. The typical sputtering parameters are: 600-V cathode voltage, 200-G solenoid field, 2×10^{-3} torr Ar pressure and 30-mA sputtering current.
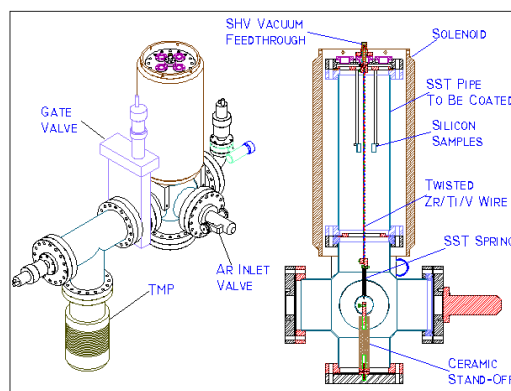

Figure 1. NEG Film Deposition System

The NEG film composition and deposition rates are measured using Rutherfield Backscattering Spectrometry (RBS). In the RBS measurements, the NEG thin film is deposited on 12 silicon samples, at various distances from the cathode for various sputtering durations, as shown in Fig.1. The RBS results from these silicon samples showed a NEG film relative composition of $Ti_{1.05}V_{1.85}Zr$ and a film deposition rate of 0.10-μm/hr on the inner wall of the 4-inch SST tube. After the RBS calibration runs,

two flanged SST tubes were coated with NEG film with a thickness of 0.6μm and 2.0μm respectively.

NEG FILM PUMPING PERFORMANCE

The vacuum pumping performance of the NEG thin films on the two SST tubes was measured using a setup depicted in Figure 2. The NEG-coated tube is connected to a TMP through a 0.25" diameter orifice. Calibrated leaks are used to introduce test gases into the system. The pressures above (P_1) and below (P_2) the orifice are measured using inverted magnetron cold cathode gauges. A residual gas analyzer (RGA) is also installed to the system. After an initial pump-down, the apparatus was baked out at 150°C for 48 hours.
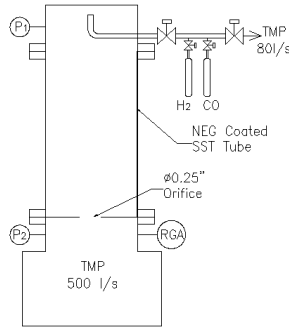

Figure 2. Apparatus for NEG Film Pumping Tests

The pumping speed and pumping capacity of the two NEG films are measured for the two test gases: hydrogen and carbon monoxide. The coated SST tube is heated to various activation temperatures (T_{act}) for durations (t_{act}) of 24-, 48- or 72-hours. The test gas is introduced into the system after the NEG-coated tube has cooled down to ambient temperature and the pressures $(P_1$ and $P_2)$ are subsequently recorded until the NEG film is fully saturated. The pumping speed of the NEG film, S_{NEG}, can be calculated from the measured P_1 and P_2 using:

$$S_{NEG} = [\overset{g}{Q}_{leak} - C(P_1 - P_2)]/P_1 , \qquad (1)$$

where $\overset{g}{Q}_{leak}$ is the leak rate of the testing gas (2.37×10^{-4} for H_2 and 1.73×10^{-5} $torr \cdot liter/sec$ for CO), and C is the gas conductance of the orifice.
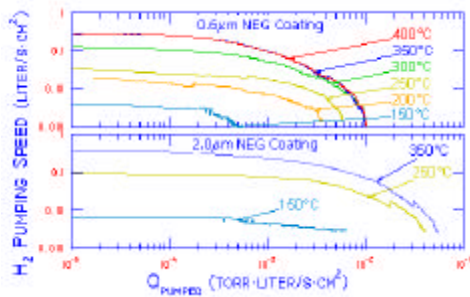

Figure 3. H_2 pumping speed vs. pumped H_2 at various activation temperatures, T_{act}, (48-hour duration).

The pumping speed measured (normalized by NEG-coated area, ~989 cm^2) for H_2 and CO are plotted as a function of the pumped gas load, Q_{pumped}, in Figures 3 and 4, at various T_{act} for the 48-hour duration. Q_{pumped} was calculated using:

$$Q_{pumped}^{CO,H_2} = \int(S_{NEG}^{CO,H_2} gP_1)dt . \qquad (2)$$

The effect of activation duration on the pumping speed and capacity for the 0.6μm NEG film activated at 350°C is shown in Figure 5. A similar trend is observed for the 2μm NEG film.
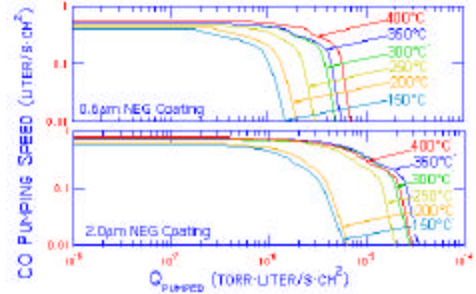

Figure 4. CO pumping speed vs. pumped CO at various activation temperatures, T_{act}, (48-hour duration).

The effect of t_{act} on the pumping is also shown in Figure 5. The pumping of CO is relatively insensitive to t_{act}. The results in Figs 3, 4 and 5 indicate that the NEG films can be fully activated at $T_{act} \geq 350°C$ with $t_{act} \geq 48$ hr.
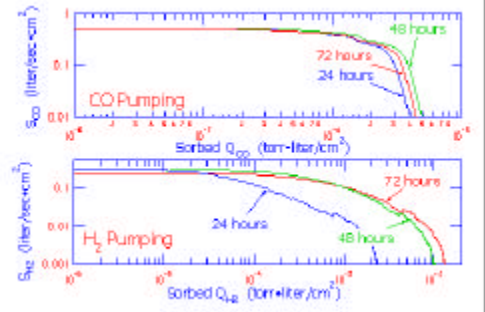

Figure 5. NEG film pumping speed at three activation durations, t_{act}, (T_{act}=350°) for the 0.6μm thick NEG film

DISCUSSION

The vacuum pumping performance of the NEG thin films can be evaluated from the initial pumping speed, S_0, (after each activation) and its pumping capacity.
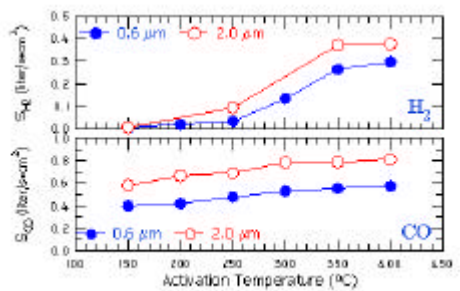

Figure 6 S_0 of CO and H_2 vs. T_{act} for both NEG films.

Using data shown in Figures 3 and 4, the dependence of S_0 of CO and H_2 on the T_{act} is plotted (Figure 6) for both the 0.6 and 2.0 μm NEG films. The S_0 of a gas depends

on the reactivity of the NEG surface to the gas, and on the effective surface area (or surface roughness). This dependence is less important for CO than for H_2. While S_0^{CO} increases slowly with increasing T_{act}, a significantly higher $S_0^{H_2}$ can be achieved with $T_{act}>250°C$. Higher S_0 is observed on the 2μm NEG film than on the 0.6μm NEG film for both CO and H_2 at tested T_{act}. This increase in S_0 on the thicker NEG film is most likely due to a corresponding increase in surface roughness. This is indicated by $S_0^{2mm}/S_0^{0.6mm}$ being nearly a constant at all T_{act} for both test gases.
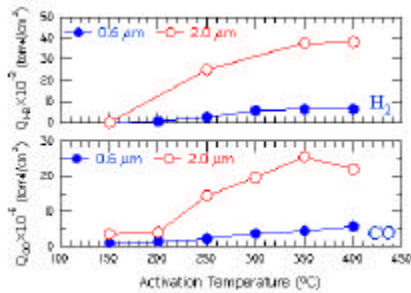

Figure 7. Pumping capacity (see test) of CO and H_2 vs. T_{act} for both NEG films.

Pumping capacity of the NEG film may be defined as the total amount of gas adsorbed (or pumped) by the film, Q_{total}, for the pumping speed to drop from S_0 to a lower value, S_f. Data in Figs. 3 and 4, and (arbitrarily) defining S_f^{CO} =0.1 l/s•cm^2 and $S_f^{H_2}$ =0.01 l/s•cm^2, gave the dependence of Q_{total} on T_{act} in Fig. 7 for CO and H_2. The results show that (1) a significant gain in pumping capacity for CO and H_2 is obtained with $T_{act}>250°C$; and (2) a much higher pumping capacity is measured on the thicker NEG film for both CO and H_2. The pumping capacity for H_2 is orders of magnitude larger than that for CO, due to different pumping mechanism for CO and H_2 [5]. CO molecules are only chemically adsorbed onto the NEG surface and this is irreversible. But the pumping of H_2 is completely reversible [5]. At RT, hydrogen molecules dissociate on the NEG surface and the atomic hydrogen then diffuses into the NEG film. The dissolved hydrogen atoms recombine on the surface and desorb as gas molecules when the NEG is heated during activation.

While the CO pumping capacity depends on surface condition of the NEG film, the H_2 pumping capacity depends on the NEG film's bulk properties, such as hydrogen solubility of the NEG film and the residual hydrogen content in the NEG film after activation.

The hydrogen solubility of the NEG films is estimated by calculating the ratio $R_{H_2}=Q_{H_2}^{max}/n_{NEG}$, in which $Q_{H_2}^{max}$ is the total H_2 pumped by a fully activated NEG film and n_{NEG} is the NEG atomic density measured by RBS. The measured maximum R_{H_2} is ~14.2% and ~15.1% for the 0.6μm and 2.0μm NEG films respectively.

To demonstrate the effect of residual H content on hydrogen pumping capacity, we measured the amount of

hydrogen desorbed during activation from a NEG film fully saturated with hydrogen, at a given T_{act}, and compared that to the amount of H_2 pumped subsequently. The results (Figure 8) clearly indicate the correlation between the H_2 desorbed from the NEG film during the activation and the H_2 pumping capacity.
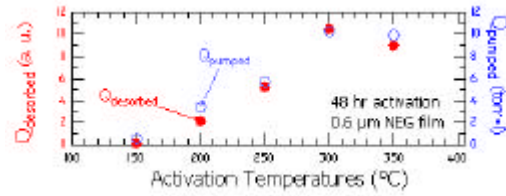

Figure 8. Amount of desorbed H_2 during activation and amount of pumped H_2 as function of T_{act}.

To illustrate the depletion of hydrogen from a saturated NEG film, deuterium was used to saturate a fully activated (72 hr at 400°C) NEG film. The measured D_2 pumping speed curve is very similar to that for H_2 shown in Fig.3. The D_2-saturated NEG film is then activated and traces of major RGA peaks during the activation are shown in Figure 9. The depletion of sorbed D_2 is evident. The observed steady H_2 desorption most likely originates from hydrogen in the substrate SST, which diffused into the NEG film and then desorbed from NEG surface at the activation temperatures.
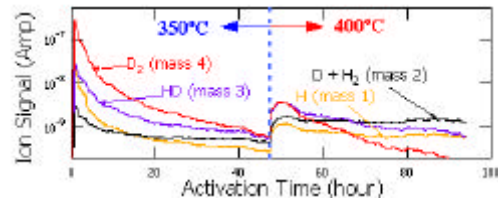

Figure 9. D_2 Desorption from NEG film during activation

CONCLUSION

TiZrV NEG thin films were deposited onto SST pipe via DC magnetron sputtering. The NEG films were characterized using RBS. Factors that affect the vacuum pumping performance of the thin films were investigated to provide 'engineering' data for vacuum system design. Vacuum pumping is observed at activation temperature as low as 150°C. Activation above 250°C will significantly enhance the pumping performance of the NEG thin films.

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