CHAPTER 5

5.1 Production of XHV using NEG coated pipes

In this chapter we present the method of achieving extreme high vacuum (XHV, pressures below $10^{-11}$ mbar) and its measurement. Achievement of extreme high vacuum in a sealed off vacuum chamber require systematic efforts. The NEG films when coated on the inner walls of a chamber help in achieving XHV in a sealed off UHV system after activation at elevated temperatures. Pumping of a sealed-off UHV system with NEG coatings has a number of benefits over other methods, such as it does not require a continuous source of power, vibration free operation, absence of magnetic field, distributed pumping in longer and narrow pipes, etc.

Several coatings of NEG were prepared on SS304L substrate using the cylindrical type DC- magnetron sputtering system to study the pumping characteristics. In this chapter a few of these will be discussed. The NEG coatings were prepared on thoroughly cleaned SS304L substrate of size 300 mm x 250 mm x 200 µm which was folded cylindrically and placed inside the cylindrical chamber. The method of preparation of the film is given in Chapter 2. The Inner surface of SS304L vacuum pipes were also coated by the same method as discussed in detail in chapter 2. The deposition time was kept 4 hrs in each case.

5.1.1 Materials for XHV

The systems being utilized in XHV allocation need to take special care and very few materials are qualified for XHV such as stainless steel, aluminium, copper and glass in early demonstrations. These materials should have been properly conditioned and achieved from highly purified form of raw materials. These materials need to be pre-baked under vacuum environment and in-situ baked. For this type of application, it is needed to prepare highly cleaned and modified surfaces. Cleaning leads to reducing the contamination levels on the
surface to an acceptable level for applications in the vacuum environment. Surface modification can involve changing the surface morphology to be more rough or smooth, changing the chemical composition of the surfaces, changing the outgassing or outdiffusion properties of the material, or changing the mechanical properties of the surface. The material needs to take special care during welding and should be free from any gas trapping regions. The materials should be free from any kinds of permeation or pumping barriers on the vacuum systems.

5.1.2 Setup for XHV measurement

Fig. 5.1 shows the schematic of the XHV set and Fig. 5.2 shows the photograph of the test set-up. In the XHV test set-up, all the vacuum pipes used were thoroughly degreased in an ultrasonic bath and later degassed in a vacuum furnace. To evacuate the chamber a Pfeiffer make turbo molecular pump combined with a dry root pump was connected via a right angle valve of size DN40. An indigenously developed triode sputter ion pump was connected to the NEG coated vacuum tube of size DN100 via a gate valve of size DN100. A vacuum tube of flange size DN100 and 300 mm long was sputtered coated using the cylindrical type magnetron sputtering with a ternary target of Ti/V/Zr in the ratio of (1:2:1). The deposition time of the film was 4 hrs and film thickness was approximately 1.5 μm which was achieved by measuring the thickness of small samples placed within the pipe at different locations. The other side of the NEG coated pipe was connected with a 6-way cross of size DN40. For measuring the 1000 mbar to $10^{-9}$ mbar pressure, a full range Pfeiffer make PBR type cold cathode gauge was connected. A Pfeiffer make Prisma residual gas analyzer was connected to the system for the measurement of partial pressures of gases. A Pfeiffer make Bayard Alpert (BA) PBR 260 gauge was all also connected to measure the pressure. Heating tape is wrapped around the surface to bake the system. During the baking of the test set up, NEG coated pipes were maintained at 100°C by air cooling of the pipe.
Temperature was controlled by a temperature controller and K-type thermocouple. A pressure of the order of $1 \times 10^{-8}$ mbar was achieved without baking the system. The set-up was then baked continuously for 36 hrs and a total pressure of $1.6 \times 10^{-10}$ mbar was achieved and measured using a residual gas analyzer (RGA) in SEM mode (1.5 kV) as shown in Fig. 5.3. The BA gauge was also in the range of lower $10^{-10}$ mbar. The measured pressure did not
reduce even after a continuous pumping with the TSIP for 3 days. The temperature of the NEG coated pipe was then raised to 180°C at a rate of 5°C/min. The temperature was kept 180°C for 24 hrs and then the heater was switched off. During activation of the NEG pipe the pressure inside the chamber increased to 5 X 10^{-9} mbar (approx.) range. The BA gauge was turned off after the activation to avoid the extra heat source. After switching off the heater the pressure of the test set-up started to decrease and goes down to less than 5 X 10^{-12} after 10 hrs as shown in Fig. 5.4.

**Fig. 5.3:** RGA measurement showing an ultimate pressure without the activation of the NEG film.

**Fig. 5.4:** Ultimate partial pressures measured after 24 hrs using the RGA after the activation of the NEG films.
5.2 Development of NEG pump with heating elements

An NEG pump was developed consisting of a strip of SS304L sputtered coated with a target of Ti-V-Zr. The size of the strip was 1200mm X 30mm X 200 μm and thickness of the coating was measured to be about 1.5μm. The photograph of the NEG pump is shown in Fig. 5.5. The strip was wound on ceramics tubes which were tightened with long bolts on a DN100 size flange. The strip was heated by passing a DC current of 10 A through a UHV compatible electrical feed-through. A K-type thermocouple was attached to the strip. An Allectra make UHV compatible feed-through was used for measuring the temperature of the strip as well as passing high current through it during the activation process. A high current/low voltage power supply was used for the resistive heating of the NEG strip.

Fig. 5.5: NEG pump developed by using coating of Ti-Zr-V films coated on SS304L substrate.
5.2.1 Activation studies of NEG pump

The NEG pump was developed and tested by mounting in a specially designed NEG test set-up Fig 5.5. The activation of the NEG strip was achieved by passing a current of 10A from a dc-power supply directly through the strip. The current was slowly increased to 10 A to elevate the temperature up to 180ºC. The strip was kept at this temperature for 24 hrs. The evolution of partial pressure of H₂ and total pressure in the XHV test set-up after activation of the NEG strip are shown in Fig. 5.6. The partial pressure of the H₂ as measured with the RGA was reduced to less than $10^{-13}$ mbar after 10 hours of activation of the NEG film at 180ºC. The total pressure inside the set up was less than $10^{-12}$ mbar. The partial pressure of CO₂ as measured by the RGA is been shown in Fig. 5.7. The partial pressure of CO₂ also decreased from $10^{-10}$ mbar to $10^{-14}$ mbar due to pumping action of the NEG strip after the activation.

![Fig. 5.6: Evolution of partial pressure of CO₂ and total pressure with time after activation.](image-url)
5.2.2 Measurement of XHV

The NEG coated surfaces under the UHV conditions need to be activated. Oxygen present in the form of superficial oxide over the films which is formed after exposing it to atmosphere is progressively reduced due to diffusion inside the bulk during the activation and the surface starts getting/sorbing the residual gas species which is referred as the pumping action of the getter materials. During the activation of the NEG, it needs to be heated either externally or internally. Activation is completed when the oxygen surface content reaches a minimum and pumping speed its maximum value.

The XHV measurement systems require special care to choose proper materials and their conditioning. All the measurement system should be properly degassed to avoid the untimely outgassing from the filament. In our XHV test set-up, residual gas analyzer is used to measure the partial pressure as well as total pressure of the system in the XHV range.

Fig. 5.7: Evolution of partial pressure of CO₂ and total pressure with time after activation.
5.3 Measurement of Secondary electron Yield (SEY)

When electrically charged particles (primary electrons) of sufficiently high kinetic energy strike a solid surface, the solid surface emits electrons. The emitted electrons are called secondary electrons (SE). The Secondary Electron Yield (SEY) is defined as the number of all the SE which are emitted per PE and as given by eq.(1), it is a three step process that includes the penetration of the PE into the target during which they produce SE through inelastic collisions with target materials, the next step is the transport of the generated SE to the surface, the third step is the escape of the SE through the surface into the vacuum. The secondary electron emission is a surface process and it is influenced by slight modifications of materials outer layers [132]. The mean escape depth of SE is of the order of 3 -5 nm [133] which is similar to the probing depth of XPS and AES. In electron spectroscopy, the electrons that contribute to the characteristic peak have a kinetic energy of more than 100 eV while vast majority of SE have energy of only a few eV. As a consequence the SE are work function influenced electrons which is not the case in AES or XPS. When measuring SEY, it is important to actually measure the yield of the material itself and not the yield from the contaminants that reside on the surface e.g. adsorbed gases and oxidation layers which can be removed by surface heating.

Secondary electron yield and surface outgassing are of special concern for accelerators and experiments undergo photo-electro diffraction [134]. Surfaces with greater secondary electron yield $\delta$ are more prone to provide multi-pacting or electron cloud instability [135]. This instability is excited by the interaction between successive charged bunches and secondary electron cloud around them multiplicatively generated by the interaction of charged bunches with the walls of vacuum pipes [136, 137]. One way to prevent these situations is to use inner surfaces with a low secondary electron yield. We have investigated the similar situation in our lab by investigating the effect of interaction of
electron beam of energy up to 2.5 keV on coated surfaces after heating at different temperatures. As the life-time of the accelerators also depends on the presence of residual gas molecules, the surfaces with high out gassing are culprit and are obstacle to increase the beam lifetime as residual molecules such as H₂ diffuses from the material due to concentration gradient. The residual molecules can also come out from the surface due to photon induced desorption [138]. These residual molecules not only act adversely with the main charge particle beams, but also reduce the life time of the beam. One of the effective solutions to this problem is to coat the whole inner wall of the vacuum pipe or chamber with non-evaporable getter (NEG) materials with an appropriate composition [139].

![Fig. 5.8: Schematic of secondary electron yield measurement system.](image)

**5.3.1 Development of SEY Measurement System**

Secondary electron yield measurement was done with an indigenously developed electron gun facilitated with inbuilt collector to collect the secondary electrons from the samples with increasing electron energy (0 -2.5keV). A current meter (Keithley make 6485) was utilized to
monitor the sample and scattered current. Schematic and actual photograph of the SEY measurement system has been shown in Fig. 5.8 and Fig. 5.9, respectively. These data basically provide secondary electron yield $\delta$ for the NEG coated samples which are calculated according to eq. (1), using the measured values of scattered and sample currents after heating the film at different temperatures. The collector voltage was set to 20V floating with the ground and base pressure was $< 10^{-9}$ mbar. The samples were prepared by sputtering technique as discussed in Chapter 2 and mounted on a 4-axes sample manipulator which is also facilitated with heating arrangements for the sample. The measurements were taken after heating the sample to different temperatures of 100°C, 140°C and 180°C for two hours.

$$SEY(\delta) = \frac{I_c}{I_c + I_s}$$

Where $I_c$ is the Collector current and $I_s$ is the sample current.

Fig. 5.9: Inside view of the secondary electron measurement set-up.
Fig. 5.10: Secondary Electron Yield (\(\delta\)) as a function of K.E. for substrate and NEG coating.

Fig. 5.11: Secondary Electron Yield (\(\delta\)) as a function of K.E. for NEG coating maintained at different temperatures.
5.3.2 Results of SEY measurement of NEG thin film on SS304L substrate

A complete quantitative description of the SE emission process consists of the SEY versus PE spectrum and the energy distribution spectrum of the emitted SE at each PE energy as shown in Fig. 5.10 which shows the reduction of secondary electron yield of NEG coating on SS304L. The reduction of SEY of NEG coating after heating it to different temperature has been also shown in Fig. 5.11. The maximum reduction in the secondary electron yield of the NEG coated film on SS304L comes after heating the film at 180ºC for two hrs even at 1 keV of primary electron energy and characteristic of the results are similar to the result published by the group CERN group [140]. This is due to dissolution of surface oxides in the bulk after the heating. The SEY measurement shows a decrease in the SEY with increasing temperature of the coated surfaces which results in lowering the multi-pacting effect in accelerators.

5.4 Summary and Conclusions

A test set-up for the XHV was developed which consists of different vacuum pumps, vacuum gauges, UHV valves. The NEG coated pipe used to create XHV by pumping the different residual gases (H₂, CO₂, H₂O, N₂, CO) was also integrated in the set-up. The activation of the pipe was done by elevating the temperature of the NEG coated pipe to 180ºC. The NEG coated pipe was kept at this temperature for about 24 hrs. After the activation, a pressure of the order of 5 X10⁻¹² mbar was achieved which was measured by using the RGA. The evolution of partial pressures of H₂ down to 10⁻¹³ mbar and 10⁻¹⁴ for CO₂ which are pumped by the NEG coating are reported. A low secondary yield of the NEG coatings which is the additional benefit of NEG coated surfaces has been discussed. The secondary electron yield exhibits the multi-pacting effect in the accelerators which adversely affects the main beam. The lowering of the SEY of NEG coating as compared to SS has been demonstrated. It has been also achieved the lowering of NEG coating after activation.