CHAPTER 4

4.1 Introduction

Coatings or bulk of NEG materials essentially act as a sorption vacuum pump due to their gettering action when activated by elevating the ambient temperature of these coatings in a sealed-off vacuum chamber. The gettering action of these coatings is due to their large solubility, and high diffusivity towards the adsorbed gases [63, 113–116]. These coatings can be used for efficiently pumping the low-aperture and micro miniature vacuum sealed devices or to construct getter pumps to complement conventional pumping devices. In addition to that, an important application of NEG materials is in large vacuum systems such as synchrotrons, particle accelerators, and colliders [113, 114, 116]. The NEG coatings on SS304L substrate were deposited using a cylindrical type DC-magnetron sputtering technique at different argon gas pressures using different targets for achieving better pumping results. As discussed earlier, an oxide layer forms on the fresh metallic surface when they are exposed to atmosphere after deposition. These coatings or thin films show their passive behaviour towards active gases unless their passive oxide is removed partially or fully in the process of activation. One method of NEG pumping is to evaporate NEG materials in both thin film and bulk form at very high temperatures make fresh film which deposited on the walls of the chamber and the fresh film starts gettering action. The other method which has been described in this chapter is the activation of the NEG films at low temperatures. In this method we sequentially test the film by heating it at different temperatures and characterize its surfaces using the XPS technique. The reduction of surface oxides shows the film is getting activated. A full reduction of the surface oxide shows the active metallic surface which is ready to chemically pump the residual gases present in an isolated chamber. The whole experiment is done under ultra high conditions. The XPS technique has been
efficiently used for this type of study. It is a sequential study which has involved a number of steps such as raising temperature of the sample at the desired level at constant rate and soaking it at this temperature for 2 – 3 hrs. After switching off the heater, it is required to achieve the required level of UHV in the chamber and allow the sample to achieve the equilibrium.

4.2 Activation studies of ternary Coating of NEG material (Ti-V-Zr) prepared by plasma arc deposition.

Before discussing the activation studies of ternary form of coating, the coating of Zr metal and binary Ti-Zr were prepared on SS304L substrate by a planer type DC-magnetron sputtering system and studies by the XPS technique. The details of the sample preparation and characterization can be found in the reference [117]. The activation temperatures of these coatings were found to be in the range from 350°C to 400°C.

The first coating of NEG materials in ternary alloy form was deposited by using a composite target of Ti$_{0.2}$V$_{0.4}$Zr$_{0.4}$ by vacuum arc evaporation at a pressure of 6 X 10$^{-5}$ mbar [118]. The target was in ingot form which was prepared by a non-consumable arc melting using a tungsten electrode in a water-cooled copper hearth, in vacuum of < 10$^{-3}$ mbar using three metals (Ti,V, Zr). The details of the preparation of the target are given in ref [118].

To study the activation process of coating of Ti-V-Zr, XPS measurements were performed in an ultra high vacuum chamber equipped with VG Scienta make sample manipulator facilitated with Tantalum heater and VG Scienta hemispherical energy analyzer facilitated with single channel analyzer detector. The analyzer worked in a constant pass energy mode with the energy resolution of about 100 meV. The excitation source MgK$_\alpha$ (1253.6 eV) was used in this experiments and take-off angle of the photoelectrons was 54.5°. The depth of the sampling was about 2 – 3 nm and the base pressure was 2 X 10$^{-9}$ mbar. The
samples were mounted on a copper holder that could be heated by a Tantalum heater. A K-type (chromel – alumel) thermocouple was attached to the sample surface to measure the sample temperature. The heating rate was kept as 5ºC/min. The samples were kept at the indicated temperature for 2 hrs and then cooled down to room temperature. While during the sample was being heated, the pressure of the chamber increased with increasing temperature. The chamber pressure did not exceed 3 X 10^{-7} mbar even after the sample was heated to 250ºC for 2 hrs. The binding energy scale was calibrated by measuring the carbon 1s peak (284.6 eV) and the core level signal of the copper holder reference before/after measuring the sample. All the spectra were normalized in intensity by using the same background-to-peak.

![Figure 4.1](image1.png)  
**Fig. 4.1:** XPS peak of Zr3d after treating at different temperatures under UHV conditions.

![Figure 4.2](image2.png)  
**Fig. 4.2:** XPS peak of V3p after treating at different temperatures under UHV conditions.
The Zr3d in oxide exhibits several peaks which can be attributed to different oxidation states of zirconium represented as Zr$^{n+}$ (where n=0 to 4), as shown in Fig. 4.1. The Zr$^{0+}$ corresponds to metallic zirconium which can be achieved after full activation of the film. The peak at 183.2 eV corresponds to ZrO$_2$ in as-deposited samples correspond to Zr$^{4+}$, i.e. zirconium is fully oxidized. On heating it losses oxygen which diffuses inside the bulk and lead to the conversion of lower oxides of Zr and then to metallic oxide Zr$^{0+}$ and metallic carbide (ZC).

The V3p exhibit several peaks which can be attributed to different oxidation states of vanadium represented as V$^{n+}$ (where n = 0 to 5), as shown in Fig. 4.2. The V$^{0+}$ corresponds to metallic vanadium which can be achieved after full activation of the film. The peak at 515 eV of as-deposited samples corresponds to V$_2$O$_4$, i.e. vanadium was not fully oxidized. On
heating it losses oxygen which diffuses inside the bulk and lead to the conversion of lower oxidation states and finally to metallic vanadium. The peak at 512.1 eV after 2 hrs of heating at 160°C corresponds to metallic vanadium mixed with VC.

The different oxidation states of Ti3p are shown in Fig. 4.3 corresponding to Ti^{n+} (where n = 0 to 4). The Ti^{0+} corresponds to metallic titanium which can exist after full activation of the film. On the other hand, the Ti3p peak at 457 eV corresponds to Ti^{4+} (TiO₂) i.e. fully oxidized state of Ti. On heating the film at 160°C its peak shifts to broad peak centred at 455 eV which corresponds to mixed value metallic Ti^{0+}, TiC and TiO as oxygen of superficial TiO₂ diffuses inside the bulk at elevated temperature. In Fig. 4.4, the shift in B.E. after heating at different temperature of the O1s peak has been shown.

In Fig. 4.5, Fig. 4.6 and Fig. 4.7, the shifting of the peak values of V3p, Ti3p and Zr 3d on account of heating has been shown which provide quantitatively the change in the B.E. of these elements in different oxide forms.

**Fig. 4.5:** Shift in the B.E. of V3p after treating at different temperatures under UHV conditions.

**Fig. 4.6:** Shift in B.E. of Ti3p after treating at different temperatures under UHV conditions.
4.3 Activation studies on ternary coatings of NEG material (Ti-V-Zr) prepared by DC-magnetron sputtering deposition (Ar pressure of $8 \times 10^{-3}$ mbar)

Thin films were deposited using a ternary target of Ti-V-Z on SS304L substrate using the cylindrical type DC-magnetron sputtering system. During the deposition, Ar pressure of $8 \times 10^{-3}$ mbar was maintained after achieving the base pressure of $2 \times 10^{-6}$ mbar. The target was made by twisting three wires of Ti/V/Zr (purity: 99.7%) of diameter (1mm) and kept at a voltage of -500V with a deposition time of 3 hrs. A magnetic field of 250 Gauss was applied using an external solenoid to increase the mean free-path of the electrons.

In Fig. 4.8, changes in the B.E. energy of the oxygen 1s peak have been shown. The peak at 530.5 eV corresponds to oxygen in Zr oxide and its suboxides, and the broad peak at 530.2 eV is due to oxygen in (Ti, V) oxides and their suboxides TiO, TiO$_2$, VO, V$_2$O$_3$, V$_2$O$_5$, etc. [119]. The oxygen peaks shifts to higher energy after heating the film which corresponds to lower metallic oxide peaks. The peak with binding energy 531.6 eV corresponds to the OH group. The surface layer of NEG film consists of all the constituent metallic oxides and hydroxides. The OH peak of air exposed films disappears in the activation of the NEG
coatings. When the films exposed to atmosphere were thermally activated the oxygen 1s peak shifts from oxides of Ti, V and their lower oxides to O peak of Zr oxide and its sub-oxides. This intends that the most oxidized films are preferentially reduced to a metallic state during the activation at 180°C for 2 hrs.

**Fig. 4.8:** XPS peak of O1s after heating at different temperatures.

**Fig. 4.9:** XPS peak of C1s after heating at different temperatures.

Fig. 4.9 shows the series of C1s spectra of the NEG film of Ti-V-Zr after being exposed to air and then heated under UHV conditions. The spectrum of the film shows that the film is composed of C-C and C-O bonds, after the film was activated at 180°C for 2 hrs. However, carbides of Ti, V and Zr which are TiC, VC and ZrC, were found in the TiVZr film after the activation due to contamination of the samples during activation by CO, CO₂. The C-C bond was also found in the activated film and remains in the further air-exposure/activation cycle.
It has been already shown that the carbides form in the subsurface region of the ZrV getter after a thermal activation of the film at 200°C [114, 115]. The formation of these metal carbides may be attributed to the low solubility (<2 at.%) of carbon in Ti, V, Zr [121]. C. C. Li et. al. also confirmed the presence of metal-oxide in the film with Raman spectroscopy technique [120].

Fig. 4.10 shows a series of Zr 3d spectra of Ti-V-Zr film after heating it at different temperatures. For the air-exposed film, the B.E. of the Zr 3d_{5/2} peak is 183.2eV, corresponding to oxidized Zr (Zr^{4+}) [118]. However, the broad peak in the Zr3d spectra after heating to 180°C corresponds to metallic Zr^0 and mixed lower oxides Zr^{n+} (n<4). This increase in binding energy is due to the presence of a larger amount of Zr oxides on the surface of the Ti-V-Zr film.

In Fig. 4.11, Ti2p_{3/2} and Ti2p_{1/2} peaks of Ti-V-Zr shown with one air-exposure/activation cycles, are at 458.0 eV and 463.2eV, respectively corresponding to those of fully oxidized Ti^{4+}[124]. After the heating cycle at different temperatures, the peaks shifted to 454.5 eV corresponding to metallic state Ti^0 and also some reduced oxides of Ti^{n+} (n<4). The TiC was observed at a higher energy of 454.7eV.

Fig. 4.12 shows a series of V2p spectra of Ti-V-Zr film with air exposed/activation cycles. The air exposed films shows broad V2p spectra with centre at 516.2 eV, and composed of V_2O_5 and its sub oxides V^{+n} (n≤ 5). Compared to the V 2p broad peak of the air-exposed film, the sharp peak of the activated film has a lower binding energy of 512.5eV after heating to 180°C. It showed the residual presence of V carbides after the activation due to CO_2 and CO [118]. O1s and C1s spectra suggest that the VC will be found in the film after the activation also.

In Fig. 4.13, the activation study of the NEG coatings exhibit a decrease in the oxygen 1s peak intensity during the activation due to a reduction of the quantity of oxygen on the
surface in the form of surface oxides. The oxygen diffuses inside the bulk of the TiZrV film at a low temperature of 150°C to 180°C due to a high diffusivity for oxygen of TiZrV produced by DC magnetron sputtering.

Fig. 4.10: XPS peak of Zr 3d shifted by almost 2.5 eV.

Fig. 4.11: XPS peak of Ti 3p shifted by almost 3.49 eV.

Fig. 4.12: XPS peak of V 3p shifted by almost 3.0 eV.

Fig. 4.13: Change in the intensity of oxygen on the NEG surface during activation.
4.4 Activation Studies of ternary alloy coating of NEG materials (Ti-Zr-Nb)

The thin films of NEG materials in ternary alloy form were prepared on SS304L substrate. The different combinations of group IVB (Ti, Zr, Hf) and V(V, Nb, Ta) were prepared on 300 x 250 x 0.2 mm³ SS substrates using the cylindrical magnetron sputtering system in Ar environment. The target was made by twisting wires (400mm long and 1mm diameter) of three metals Ti, Nb and Zr in the ratio of 1:2:1. The target and substrate were cleaned thoroughly in an ultrasonic bath with soap solution, acetone and methanol. After the ultrasonic bath, both were dried by purging high speed dry nitrogen from the air gun. The sample cleaning is essential for good adhesion and purity of the deposited film. A base pressure of about 2x10⁻⁷ mbar was achieved before the deposition and Ar pressure of 1.2 x10⁻² mbar was kept during deposition. The deposition time was 3 hrs with the deposition rate of about 3 Å/s. After the deposition, the film was exposed to air and then placed inside the XPS chamber with a load lock arrangement. The VG Scienta make sample manipulator facilitated with heating arrangement was used for the sequential study of activation. The sample was heated between 100°C and 300°C at rate of 5°C/min for two hours. During heating the pressure of the UHV chamber was increased from 10⁻⁹ mbar to 10⁻⁷ mbar. The sample was then cooled to the room temperature. XPS spectra of the sample were recorded using MgKα X-ray source after heating at each temperature.

Fig. 4.14 shows the reduction of Zr3d peak that started from the oxide peak Zr⁴⁺ at 183 eV to Zrⁿ⁺ (n < 4) after heating the sample to 250°C as the peak was broaden. After 300°C, the Zr3d peak was clearly reduced to 179 eV which is a metallic peak of Zr⁰ of zirconium.
Fig. 4.15 shows the reduction of Nb3d peak from the oxide Nb\(^{5+}\) at 208 eV to Nb\(^{n+}\) (n<5) after heating to 250°C as the peak clearly shifted to 204 eV corresponding to metallic Nb\(^0\). After 300°C the Nb3d peak clearly shows a split in to 3d\(_{5/2}\) and 3d\(_{3/2}\).

In Fig. 4.16 oxygen 1s peak is shown after heating the sample at different temperatures from 100°C to 300°C. The air exposed film showed a broad 1s peak of oxygen in the oxide form at 529.5 eV and 530 eV corresponding to oxides of Ti, Nb and oxide of Zr. The small peak at 532 eV corresponds to CO. After heating the sample the CO peak grows indicating the formation of carbon monoxide at the surface. This CO peak disappears after heating to 250°C and a single peak of oxygen remains. In Fig. 4.18, the intensity of the oxygen 1s peak in oxides also goes down after activation which reflects the dissolution of superficial surface oxides inside the bulk. To monitor the oxygen intensity, each curve is normalized. The activation temperature of this NEG coating (Ti-Nb-Zr) was found to be about 250°C. The full activation has been achieved at 300°C for 2 hrs of activation, as shown in Fig. 4.17.

![Fig. 4.14: XPS peak of Nb3d\(_{5/2}\) after heating the Ti-Nb-Zr film at different temperatures.](image)
Fig. 4.15: XPS peak of Zr3d5/2 after heating Ti-Nb-Zr film at different temperatures.

Fig. 4.16: XPS peak of O1s after heating the Ti-Nb-Zr film at different temperatures.
The surface plays an effective role for the adsorption of the residual reactive gases and adsorption sites are depended on the quality of the film surface. Presence of the more number of grain boundary leads to the bulk as well as surface diffusion. The columnar growth is preferable as it leads to increased number of adsorption sites. The ternary coating of Ti-Nb-Zr was studied by secondary ion mass spectra (SIMS) results are shown in Fig. 4.19, Fig. 4.20 and Fig. 4.21. The SIMS can also be used for the activation studies of the NEG coatings [122]. The spectra reflect the presence of all the constituents elements and their oxides in almost same chemical composition as revealed by EDAX measurements that are given in Table 4.1.

**Table 4.1.** Atomic fractions of the Ti-Zr-Nb film deposited on SS304L.

<table>
<thead>
<tr>
<th>Element</th>
<th>Atomic fraction(EDX)</th>
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<tbody>
<tr>
<td>Ti</td>
<td>20%</td>
</tr>
<tr>
<td>Nb</td>
<td>55%</td>
</tr>
<tr>
<td>Zr</td>
<td>25%</td>
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</table>
4.5 Activation studies of different ternary Coatings of Ti-V-Zr prepared by DC-magnetron sputtering deposition (Ar pressure of 1.1 X10^{-2} mbar)

Coatings of NEG of ternary alloy Ti-Zr-V were deposited using the DC magnetron sputtering technique at Ar pressure of 1.1 X10^{-2} mbar. The activation studies of the coating were carried out using the XPS technique under UHV environment. The sample was heated in-situ to desired temperatures in the range from 140°C to 200°C for 3 hrs at a constant rate. The XPS spectra were recorded after every heating.
All elementary XPS peaks are shifted by about 2 eV to lower BE for Zr(3d₅/₂) as shown in Fig. 4.22. These lower B.E. peaks correspond to reduction of higher to lower Zr oxide states due to diffusion of oxygen present in the form of oxide from the surface into the bulk during activation.

In Fig. 4.23, Ti (2p₃/₂) peak of an as-deposited sample shifted by about 2 eV to a lower B.E. after heating the sample to 180°C. It corresponds to the lower oxide state of Ti on the surface that forms after the reduction of surface oxide due to the diffusion of oxygen atoms into the bulk during activation.

In Fig. 4.24, vanadium(V2p₃/₂), B.E. peak shifted by about 4 eV to lower B.E. after heating at 180°C which is a metallic state of V. The vanadium is completely converted to metallic state after the diffusion of all oxygen atoms in to the bulk.

The activation of the ternary Ti-V-Zr film was seen to proceed by the subsequent transition of oxides (TiO₂, ZrO₂ and V₂O₃) to lower suboxides of Ti, Zr and V mixed with metallic Ti, Zr and V phases after 180°C. This oxide reduction leads to the formation of active surface for CO and CO₂ to form carbides and oxide. H₂ dissociated over the active NEG surface and dissolved in the bulk as solid solution. These effects result in the lowering of the pressure inside the chambers.

The XPS studies exhibited the lowest achievable activation temperature that lies in the range from 160°C to 180°C for 3 hrs of heating of Ti-Zr-V films. The Ti-V-Zr films can be used as NEG to achieve lower pressures in extreme high vacuum applications.

In Fig. 4.25, the XPS of C1s peak shows that the film is composed of C-C and C-O bonds, after the film was activated at 180°C for 2 hrs. However, a residual presence of carbides of Ti, V and Zr were found in the TiVZr film after the activation. In Fig. 4.26 and Fig. 4.27, the SIMS spectra of as-deposited NEG film are given which show the presence of Ti⁺, V⁺. Zirconium is present in the form of both Zr⁺ as well as ZrO⁺ oxide.
Fig. 4.22: XPS peak of Zr3d after heating it at different temperatures.

Fig. 4.23: XPS peak of Zr3d after heating it at different temperatures.

Fig. 4.24: XPS peak of Ti3p after heating it at different temperatures.

Fig. 4.25: XPS peak of C1s after heating it at different temperatures.
Fig. 4.28 shows the change in the B.E. energy of the oxygen 1s peak. The peak at 530.5 eV corresponds to oxygen in Zr oxide and its suboxides, and the broad peak at 530.2 eV is due to oxygen in oxides of Ti, V and their suboxides TiO, TiO₂, VO, V₂O₃, V₂O₅ etc. [119]. The oxygen peaks shift to higher energy after heating the film which correspond to lower oxide states of metals. The peak with binding energy 531.6 eV corresponds to the OH group. The surface layer of NEG film is constituted with metallic oxides and hydroxides. The OH peak of air exposed films disappears in the activation of the NEG coatings. When the films exposed to atmosphere were thermally activated the oxygen 1s peak shifts from oxides of Ti, V and their lower oxides states to O peak of Zr oxide and its sub-oxides. This indicates that most of the oxidized on the air-exposed films are preferentially reduced to a metallic state during the activation at 180°C for 3 hrs.

Fig. 4.26: SIMS spectra of as deposited film showing Ti⁺ and V⁺.

Fig. 4.27: SIMS spectra of as-deposited film showing Zr⁺ and ZrO⁺.
4.6 Crystallographic structure of NEG coating of Ti-V-Zr

Crystalline structure information of the NEG coating was studied using the grazing incident X-ray diffraction. Samples of NEG films on SS304L were studied on an XRD beam line at Indus-2 synchrotron radiation source. This technique works on the Bragg’s law as described in Chapter-3 [110, 123- 128]. For this purpose, a monochromatic beam of X-rays of 17 keV was used as a source. The beam of X-rays was made to incident at a grazing angle of 3º for maximum absorption by the thin film. The diffraction pattern shown in Fig. 4.29 revealed the peaks due to the film as well as those from the substrate. Using the JCPDF software, it was
found that the film had polycrystalline grains and all the constituent elements of the film were present in metallic as well as metal-oxide phases.

4.6.1 Calculation of crystallite size

Under the assumption of homogeneous single phase and of equiaxed crystal grains, Debye Scherrer formula can be applied to calculate the average crystallite size of the polycrystalline film [111, 130, 131]. The formula is given as:

$$\omega = \frac{k \cdot \lambda}{\Delta_c(2\Theta) \cdot \cos \Theta}$$

where, $\lambda$ is the wavelength of the source, $2\Theta$ is the angular position of the recorded peak in radians; and $\Delta_c(2\Theta)$ is the full width at half maximum of the peak at position $2\Theta$ in radians, $k$ is the shape factor which is (k = 0.94) for cubic crystalline and cubic geometry shape. The constant k has been determined that varies between 0.89 and 1.42 [131]. The calculated crystallite size is found to be 35 nm for a film with an average composition of Ti$_{21}$V$_{55}$Zr$_{24}$.

![XRD pattern of NEG thin film of Ti-Zr-V recorded using an XRD beamline on Indus-2 synchrotron radiation source.](image)

Fig. 4.29: XRD pattern of NEG thin film of Ti-Zr-V recorded using an XRD beamline on Indus-2 synchrotron radiation source.
4.7 Elemental composition of NEG thin films

4.7.1 Energy Dispersive X-ray spectroscopy (EDX)

In-depth stoichiometry of the NEG coatings was measured by EDX spectroscopy which works on the principle of the emission of characteristic X-ray radiation from the sample as a result of the bombardment of the primary beam of electrons on the samples. It is elaborately described in the references [123, 124]. The constituent elements are identified with the help of well known Mosley’s law that relates the wavelength of the each observed spectral line to a specific element through the relationship:

$$\frac{1}{\lambda} = k.(Z - \sigma)^{\frac{1}{2}}$$

where, Z is the atomic number of the material, σ is the screening constant and k is the for each spectral line series, λ is the x-ray wavelength [125]. This technique is good for high Z materials and light elements are poorly detected due to their low fluorescent yield as well as their low energy of characteristic X-ray which is reabsorbed by the sample itself.

EDX measurement were performed in a Tescan Vega make Scanning Electron Microscope. The analyzed depth of the film is the order of 1000 nm. The evaluated thin film compositions of different NEG films which were studied in the present work are given in Table 4.2.

**Table 4.2:** Chemical composition of different NEG films by EDX spectra.

<table>
<thead>
<tr>
<th>Preparation Technique</th>
<th>NEG film</th>
<th>Stoichiometry</th>
<th>Activation temperature</th>
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<tbody>
<tr>
<td>Vacuum Plasma Arc</td>
<td>TiVZr</td>
<td>Ti$<em>{23}$V$</em>{47}$Zr$_{30}$</td>
<td>160°C</td>
</tr>
<tr>
<td>DC-magnetron Sputtering</td>
<td>TiVZr</td>
<td>Ti$<em>{21}$V$</em>{55}$Zr$_{24}$</td>
<td>180°C</td>
</tr>
<tr>
<td>DC-magnetron Sputtering</td>
<td>TiNbZr</td>
<td>Ti$<em>{20}$Nb$</em>{25}$Zr$_{25}$</td>
<td>250°C</td>
</tr>
<tr>
<td>DC-magnetron Sputtering</td>
<td>TiVZr</td>
<td>Ti$<em>{28}$V$</em>{45}$Zr$_{27}$</td>
<td>180°C</td>
</tr>
<tr>
<td>RF-magnetron Sputtering</td>
<td>TiVZr</td>
<td>Ti$<em>{48}$V$</em>{20}$Zr$_{32}$</td>
<td>200°C</td>
</tr>
</tbody>
</table>
4.8 Summary and Conclusions

To summaries, the activation studies were carried out on different NEG coatings prepared by vacuum plasma arc deposition and DC-magnetron sputtering technique. The EDX measurements provided the chemical composition of the deposited films. The activation studies are done using in-situ XPS techniques under UHV conditions. The results revealed the activation temperatures of these NEG films range from 160°C to 250°C. The NEG film of chemical composition Ti\textsubscript{23}V\textsubscript{47}Zr\textsubscript{30} which was deposited by the vacuum plasma arc deposition using a composite target of Ti-V-Zr exhibited an activation temperature of 160°C after 2 hrs of heating. The second NEG film of chemical composition Ti\textsubscript{20}Nb\textsubscript{55}Zr\textsubscript{25}, prepared by the DC-magnetron sputtering showed the activation temperature of 250°C after 2 hrs of heating. The other NEG film of chemical composition Ti\textsubscript{28}V\textsubscript{45}Zr\textsubscript{27}, prepared by the DC-magnetron sputtering showed the activation temperature of 180°C after 2 hrs of heating. The NEG film prepared by RF-sputtering and chemical composition Ti\textsubscript{48}V\textsubscript{20}Zr\textsubscript{32} showed the activation temperature of 200°C. The GIXRD of the NEG film on SS304L substrate showed the polycrystalline nature of the film with an average grain size of 35 nm. The activation studies of the film using the XPS showed that the reduction of higher oxides of Ti, Zr and V into lower oxide and then to metallic, revealed the activation starts at 160°C for this film.