

T.3: Non-evaporable getter coating for UHV application in accelerators at RRCAT

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Abstract

Non-evaporable getter (NEG) thin film is an advanced vacuum pumping solution to achieve very low pressure down to extreme high vacuum ($<10^{-11}$ mbar) condition in a vacuum chamber at room temperature. The need to provide ultra-high vacuum (UHV) conditions, along the low-conductance beam vacuum chambers in order to assure the required beam lifetime, has prompted the large scale deployment of NEG thin film coating in modern particle accelerators for high energy physics and synchrotron radiation sources. This article summarizes the activities and technological developments related to NEG thin film, for accelerator UHV applications, in recent years at RRCAT.

1. Introduction

UHV pressure of $\sim 1 \times 10^{-9}$ mbar or lower is required in beam vacuum chambers of electron storage rings for longer beam lifetime. Multi-bend achromat (MBA) lattice of low emittance fourth generation synchrotron radiation source (SRS) requires densely populated magnets with very small pole gap to provide desired high strength magnetic fields. This type of compact and dense lattice design leads to considerable reduction of cross-section dimensions of beam vacuum chambers having very low gas conductance. The gas source is uniformly distributed along the beam vacuum chambers and hence distributed pumping arrangement at very close intervals is required to achieve the desired average pressure along the beam vacuum chambers in unit cell lattice. Pumps located at regular distances from each other give rise to a parabolic pressure profile with minima at the pump location. The non-availability of drift space between magnets precludes installing conventional lumped UHV pumps at very close intervals. In this scenario of highly compact lattice design, attaining operating UHV pressure condition, with conventional lumped UHV pumps, becomes very difficult and to overcome this challenge, vacuum scientists at CERN developed non-evaporable getter (NEG) thin film coating, as linearly distributed pumping solution, for the low conductance beam vacuum chambers of large hadron collider (LHC) in the late 90s' [1] and subsequently this technology has been used in several synchrotron light sources like ESRF-France, SOLEIL-France, MAX-IV-Sweden and SIRIUS-Brazil, etc. at large scale to produce a uniform pressure profile. Along with achieving very low ultimate pressure, NEG thin film provides large pumping speed and sorption capacity for the gases present in UHV systems, i.e., H_2 , CO, O_2 , and H_2O apart from N_2 & O_2 in case of leaks, without conductance limitation. In addition to simplification of vacuum chamber design, it also provides low photon induced desorption (PID) yield and secondary electron yield (SEY) for accelerator application. It doesn't need any extra space, electric power, insulation and feedthroughs for its functioning once activated.

Sensing the inescapable need of NEG thin film pumping technology for Indus upgradation and upcoming low emittance high brilliance synchrotron radiation source, RRCAT initiated development of indigenous NEG coating technology way back in 2009.

2. Materials for NEG thin film

Transition metals are well known for their high reactivity with gases and have been used as getter materials in sealed-off vacuum devices since long time. Getter materials pump reactive gases by chemisorption and hydrogen by diffusion. In order to facilitate pumping action getter material requires clean surface free from oxide passivation layer. By heating in vacuum to a high temperature, the native oxide layer of such a film is dissolved into the bulk allowing chemisorption of most of the gases present in vacuum systems at room temperature. This thermal treatment process is called 'activation'. Once activated, the pumping starts and continues till saturation of getter material.

For accelerator vacuum application, it is desired to have NEG film with activation temperature compatible with maximum temperature allowed for normally used construction materials of UHV chambers like stainless steel (SS), copper and aluminium, etc. Alloying of different transition metals like: titanium (Ti), zirconium (Zr) and vanadium (V) is done to achieve the low activation temperature in the range of 180 to 250 °C compatible with vacuum chamber construction materials [2]. The dissolution of native oxide is possible at such temperatures only if the transfer of oxygen in the solid solution is thermodynamically possible and the oxygen diffusion rate in the film is sufficiently fast. It turns out that the key parameters for the choice of NEG coating materials are: high oxygen solubility limit in solid solution and the oxygen diffusivity in the film. Nano-crystalline NEG film of Ti-Zr-V with $\sim 1 \mu m$ thickness (typical) and activation temperature of 180 °C for 24 hrs. is the state-of-the-art [3] and have been deployed in aluminium beam chambers of many accelerators in operation around the world including Indus-2. Ti and Zr facilitate high oxygen solubility limit, while V supports reduction of grain size, as a result, increase transport of oxygen through the grain boundaries. Development of accelerator compatible low activation temperature NEG alloy film, with good vacuum performance characteristics like pumping speed and sorption capacity, requires stringent control of chemical composition and morphology of the deposited film.

3. Infrastructure development for NEG coating

3.1 Vertical magnetron sputtering deposition system

Glow discharge sputtering is the most commonly used thin film technique for coating a wide range of materials for various application. This process allows co-sputtering from composite cathode, which is desired for developing alloy thin film coating for accelerator application. Cylindrical DC magnetron sputtering is the obvious suitable configuration for coating tubular vacuum chambers normally used in accelerator vacuum systems.

In view of the requirement of long UHV chambers for insertion devices in Indus-2 and for future high brilliance synchrotron radiation source, a cylindrical DC magnetron sputtering based large coating system has been indigenously developed and installed in class 10000 clean room at RRCAT. The overall size of the system is 1300 mm (length) x 1200 mm (width) x 5500 mm (height). Vacuum chambers with cross-section dimension falling within uniform solenoid field of diameter 290 mm and length up to 3500 mm can be accommodated for thin film coating in this setup. Photograph of the installed coating system is shown in Figure T.3.1.



Fig. T.3.1: Photograph of vertical coating setup.

Process & instrumentation (P&I) diagram of the coating system is shown in Figure T.3.2.

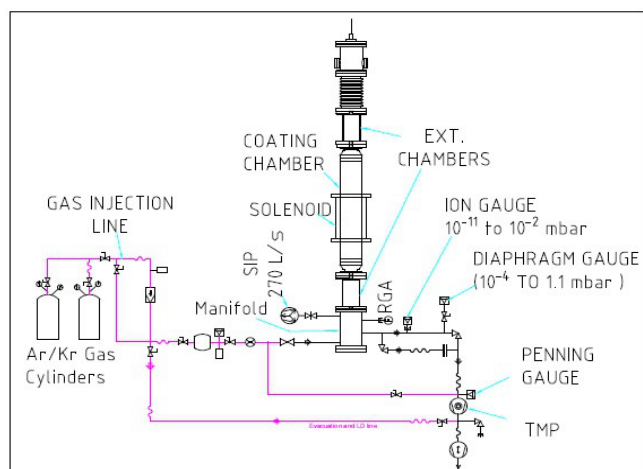


Fig. T.3.2: P&I diagram of NEG coating setup.

Brief details of various sub-systems of this coating setup are described below:

3.1.1 Vacuum system

All metal UHV system comprising of sputter ion pump, Turbomolecular Pump (TMP) station with dry backing pump for initial roughing & baking, gate valve, Bayard Alpert Gauge (BAG), Residual Gas Analyzer (RGA), Capacitance Diaphragm Gauge (CDG), auxiliary chambers and port for connecting Mass Spectrometer Leak Detector (MSLD) for helium leak detection. Ultimate vacuum performance of vacuum chambers (pre- and post-coating) is assessed along with RGA spectrum of residual gases.

3.1.2 Gas dosing system

Supply of inert gas atoms (Ar, Kr) for the ionization process is made through gas dosing system comprising of gas cylinder, dew point sensors, in-line purifier, ultra-pure buffer gas reservoir, leak valves and electro-polished SS tubing, valves, pirani gauge and mass flow meters.

3.1.3 Solenoid

Water cooled 360 mm inner diameter (ID) and 1000 mm long solenoid capable of generating axial magnetic field upto 630 Gauss is installed to increase the ionization efficiency, improving stability and allowing lower discharge pressure. This solenoid provides uniform axial magnetic field within 290 mm cylindrical zone.

3.1.4 Solenoid movement mechanism

In order to coat longer chamber, solenoid shall be moved and parked at variable heights. To facilitate this, lead screw based linear movement mechanism capable to carry solenoid weighing 300 kg is installed. Stroke length and speed movement are 3750 mm and 300 mm/min., respectively.

3.1.5 Power supplies

Bipolar DC regulated constant voltage – constant current (CV-CC) power supply of 600 V and 5.5 A (TDK-Lambda make) rating is installed to facilitate sustained discharge of process gas for sputtering. During the glow discharge cleaning, substrate is kept at ground potential and target is at positive potential, whereas for coating, target is kept at negative potential and substrate at ground potential. At the time of coating, power supply is used in CC mode for constant current discharge. 200 A, 50 V indigenously developed, DC constant current controlled power converter, is deployed for powering solenoid coils in NEG coating facility.

3.1.6 Bakeout and activation controller

Bakeout of vacuum system is carried out for desorption of water vapour during pump down for achieving UHV condition. Depending on the construction material, rate of temperature rise/fall, flat top temperature & dwell time are specified for reduction of specific outgassing rate of vacuum chamber material. Along with bakeout, activation of NEG film of coated chamber is also pursued for initiating pumping action. To accomplish the combined bakeout and activation cycle, temperature control system was developed and installed. Design details of the temperature controller is reported in RRCAT Newsletter, Vol. 30 Issue 1, 2017.

Main features of graphical user interface (GUI) are listed as follows:

- Auto/Manual set point provision with dual ramps & Dwell phases.
- Supports nine auto set point profiles with multi-plot view.
- Supports six Temperature Control Units (TCU) each of 8 channels.
- Supports 8 pressure monitoring gauges
- Provision for keeping uniform temperature between channels.

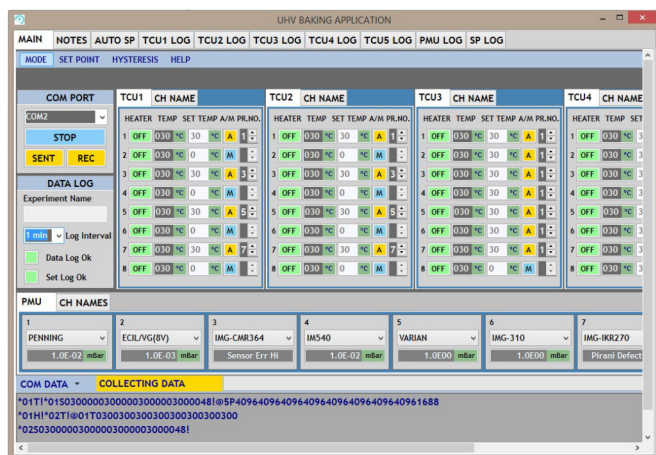


Fig. T.3.3: Screenshot of software showing overall GUI.

- Provision for user notes, channel names & cycle name.
- Status displayed communication status, log status, serial data, heater status, etc.
- Auto recover setting option after power failure.

Screenshot of software showing overall GUI of temperature controller is shown in Figure T.3.3.

3.1.7 Data acquisition and process control system

For data logging of various process parameters in NEG coating system, compatible hardware & software were developed. The controller is based on LPC1768 processor. This unit supports analog & digital outputs and serial communication. Some of the instruments are directly interfaced over RS232/ RS485 serial bus to PC.



Fig. T.3.4: Screenshot of data logging of various parameters in NEG coating system.

This setup logs the data of various instruments providing overall supervision & control (GUI shown in Figure T.3.4). The instruments are listed below:

- Discharge power supply.
- Solenoid power supply.
- TMP controller TwisTorr 304 FSAG.
- Vacuum gauge controller TPG 262 for vacuum gauge heads CMR361 & 364.
- BA Gauge controller: Agilent make.
- Dew point and pressure transmitter DPT146
- Mass flow meter.

The setup has been used in several coating cycles successfully.

3.2 Horizontal magnetron sputtering deposition system

Several experimental studies are required to be carried on test samples to optimize the NEG coating process parameters prior to deployment of NEG coating on actual vacuum chambers. To meet this requirement, a small horizontal magnetron sputtering deposition system was developed in lab [4]. Photograph of this system is shown in Figure T.3.5. Vacuum chambers of size upto 400 mm length and 200 mm diameter can be accommodated in this setup for NEG coating.

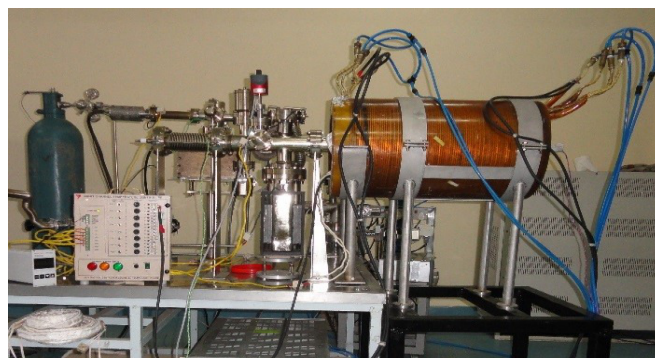


Fig. T.3.5: Horizontal coating setup.

3.3 Ultimate vacuum test setup

An ultimate vacuum test facility, suitable for testing the ultimate vacuum performance of NEG coated chambers, was setup in the lab. Schematic diagram of the test setup is shown in Figure T.3.6. The test setup consists of 270 l/s sputter ion pump (SIP) and 500 l/s titanium sublimation pump (TSP) connected in series. Both the pumps are separated from coated chamber through an orifice [5]. One extractor gauge (LEYBOLD, IONIVAC IM 540) capable to read pressure down to 2×10^{-12} mbar is installed at the extreme end of the NEG coated chamber to measure total pressure in ultimate vacuum condition after NEG activation. BAG (Varian) and RGA (Extorr) are installed at the conventional pumps side to measure the total pressure and partial pressure of gases, respectively in ultimate vacuum condition.

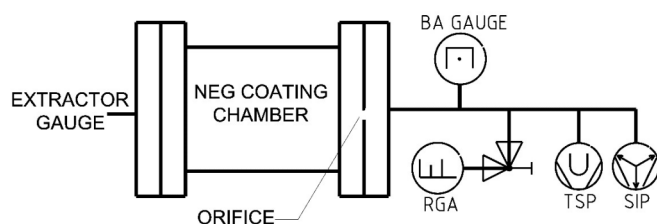


Fig. T.3.6: Schematic of the ultimate vacuum testing setup for NEG coated vacuum chamber.

4. NEG coating of stainless steel vacuum chamber and results

Multiple SS316L chambers, having 100 mm diameter and 400 mm length were coated with NEG film using axially placed single target made of intertwined wires of Ti, Zr & V of 1 mm diameter each in 1:1:1 ratio and their vacuum performance were tested. Best ultimate vacuum of 7×10^{-12} mbar was achieved for the chamber activated at 300 °C [6].

Pumping speed measurement of two chambers, namely CH#E & CH#F coated at 150 °C and 250 °C temperature, respectively, were carried out for CO & H₂ gases with varying activation temperatures. Results of pumping speed measurement data is presented in Figure T.3.7. Higher pumping speed was achieved for the chamber CH#E, which was coated at 150 °C substrate temperature [7].

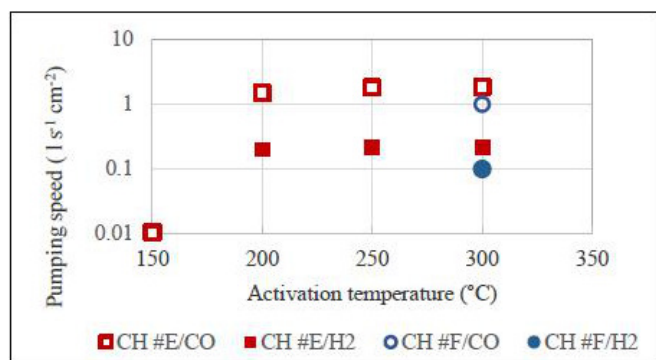


Fig. T.3.7: Pumping speeds of NEG coated SS chambers.

4.1 Morphology of coated NEG film

The morphology of coated NEG film of CH#E as scanned by Atomic Force Microscopy (AFM) is shown in Figure T.3.8, which exhibits rough appearance. Typical chemical composition of grown film on SS substrate is: Ti35%, -Zr22%-V43% (atm%). V percentage is higher due to its higher sputtering yield. Measured thickness of coating was found to be in the range of 1.5 μm to 1.65 μm.

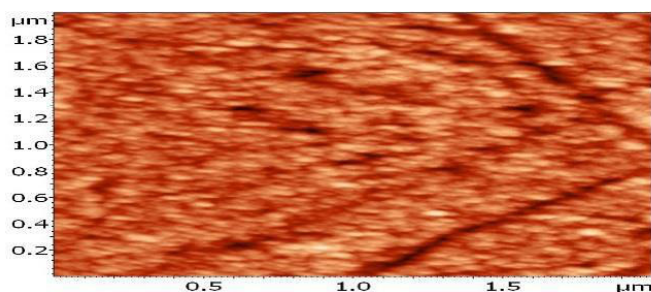


Fig. T.3.8: AFM 2D image of NEG film grown on SS.

5. NEG coating of aluminium alloy vacuum chamber and results

Aluminium and copper are widely used construction materials of beam chambers in modern high brilliance synchrotron radiation sources. Indus-2 beam chambers are made of aluminium alloys. Expertise for their design, UHV compatible welding, aluminium metal sealing technology [8], cleaning, vacuum testing, installation and maintenance in the storage ring exists in-house. Activation of NEG coating on aluminium chambers requires heating to minimum temperature of 180 °C for 24 hrs. Indigenously developed AA6063-T6 extrusion chamber with AA6061-T6 flange and diamond profile metal seal of AA6063-T5 has well proven capability to sustain 180 °C with 24 hrs. of multiple heating cycles without any leakage issue.

Three undulators, deployed in Indus-2, are housing imported NEG coated aluminium chambers and another two upcoming insertion devices will also require NEG coated aluminium beam chambers as part of upgradation plan. Upcoming 4th generation synchrotron radiation source (SRS) will require large number of NEG coated beam chambers. Working undulators, in Indus-2, require spare NEG coated chambers in ready stock for replacement in case of irreversible damage of NEG coating due to occurrence of large leakage and air rush inside during activation cycle. Keeping in view of the above inescapable developmental need, recently one NEG coated AA6063-T6 vacuum chamber, with internal aperture of 81 mm x 17 mm and 2700 mm long and diamond profile Al metal sealing, was developed indigenously [9] and its vacuum performance was assessed. NEG coating of this long chamber has been carried out in vertical magnetron sputtering system.

Prior to NEG coating of actual long chamber, experimental coating runs were carried out to optimize coating process by simulating the shape and size of actual chamber with dummy chamber containing sample holding cage. Sample holding cage is having internal dimension of 17 mm x 81 mm (same as internal aperture of spare undulator chamber). Glass strip (Figure T.3.9) masked at regular intervals to create the steps for thickness measurement were placed inside the vacuum chamber. Coating was performed in the vertical coating setup. Due to shorter solenoid of 1 m length, coating of entire chamber was carried out in four steps by shifting the solenoid positions. Two targets were installed inside the chamber for getting the uniform coating.

Thickness of NEG film on glass samples were measured by stylus profilometer. The measured film thickness uniformity achieved is within +/-30% range in longitudinal direction as shown in Figure T.3.10 [10]. Average thickness of coating is 1.07 μm.



Fig. T.3.9: NEG coated long glass strips.

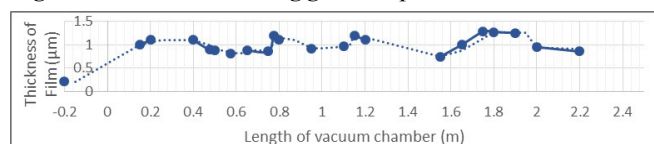


Fig. T.3.10: Measured coating thickness along long coated glass strip.

Subsequent to process optimization for uniform thickness coating of long chamber, Ti-Zr-V NEG film was deposited onto the interior wall of 2700 mm long aluminium chamber following the optimized process using double target wires of Ti-Zr-V and Kr as process gas. Coating process parameters are: pressure of 8×10^{-3} mbar, discharge voltage of -600 V, discharge current of 150 mA, magnetic field of 250 G, substrate temperature of 100 °C. Glass and aluminium test samples, placed in extension chamber, were also coated simultaneously

with the main chamber for coating surface characterisation. Chamber cross-section sketch with location of target wires is shown in Figure T.3.11.

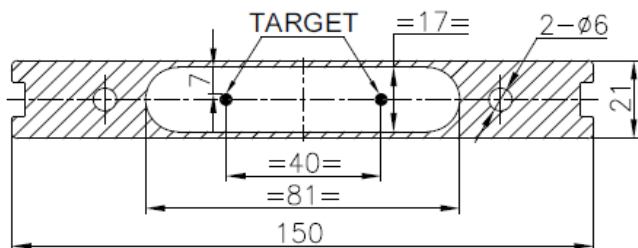


Fig. T.3.11: Schematic of the cross-section of aluminium chamber with location of targets.

5.1 Ultimate vacuum performance results

The vacuum performance of the NEG coated chamber was studied by means of ultimate vacuum testing and RGA analysis. After completion of coating cycle, coated chamber was activated in-situ in coating setup and ultimate pressure of 2.4×10^{-10} mbar was achieved. Most abundant gases H_2 and CO with partial pressures of 7×10^{-10} mbar and 1.2×10^{-11} mbar, respectively are observed. In coating setup, surface area of uncoated components is much larger as compared to coated chamber surface area and hence higher outgassing load from uncoated surfaces is limiting the ultimate vacuum performance of the coated chamber. In view of this, the ultimate vacuum performance of coated chamber was tested on separate ultimate vacuum test setup. Photograph of ultimate vacuum test setup integrated with NEG coated 2700 mm long aluminium chamber is shown in Figure T.3.12. The typical temperature vs time profile during the baking and NEG activation cycle is shown in Figure T.3.13. Indigenously developed polyimide insulated thin film heaters were mounted on the chamber for bake out and activation using in-house developed temperature controller. Initially, SIP and TSP were baked for 24 hrs. at 220 °C, RGA at 150 °C and NEG coated chamber temperature was maintained at 80 °C to avoid the poisoning of coated chamber. This was followed by activation at 180 °C for 24 hrs. After post activation cool down and subsequent pumping for 24 hrs., an ultimate vacuum of 2×10^{-11} mbar was achieved at the end of NEG coated chamber side, whereas 6.6×10^{-11} mbar was achieved towards the SIP and TSP combination pumps side. Pressure gradient clearly shows the NEG coated chamber behaving like a pump facilitating better vacuum towards the NEG coated chamber. Partial pressure of residual gases after NEG activation is shown in Figure T.3.14, showing Kr gas partial pressure reduced to below 10^{-12} mbar.

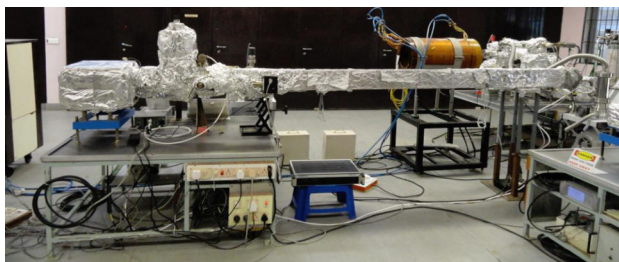


Fig. T.3.12: 2700 mm long coated aluminium chamber integrated with ultimate vacuum test setup.

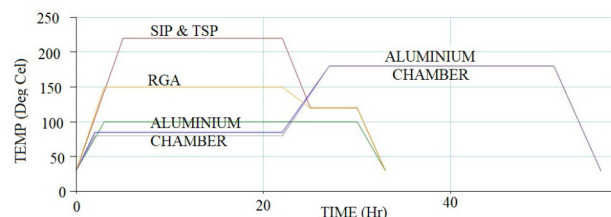


Fig. T.3.13: Temperature profile for baking and NEG activation.

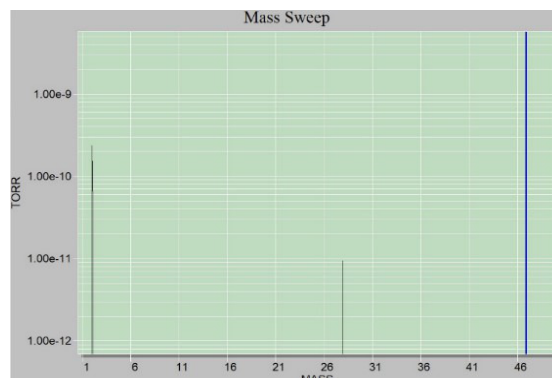


Fig. T.3.14: RGA spectra after NEG activation.

5.2 Surface characterization of coated NEG film

Small test coupons simultaneously deposited along with long chamber were studied for surface characterisation as described below:

5.2.1 Morphology of coated NEG film

The aluminium samples of 10 mm x 10 mm size placed inside the extension chamber were coated simultaneously with the 2700 mm length aluminium chamber. The samples were studied for film morphology using secondary electron microscopy (SEM). Temperature of aluminium sample during coating was also maintained at 100 °C, which was same as main chamber. Figure T.3.15 shows the cauliflower type structure of coated film as observed by SEM imaging. This typical film structure specific to aluminium substrate has been reported to provide higher absorption capacity as compared to the dense surface [11]. The effect of substrate temperature on deposited film morphology is mentioned in reference [12].

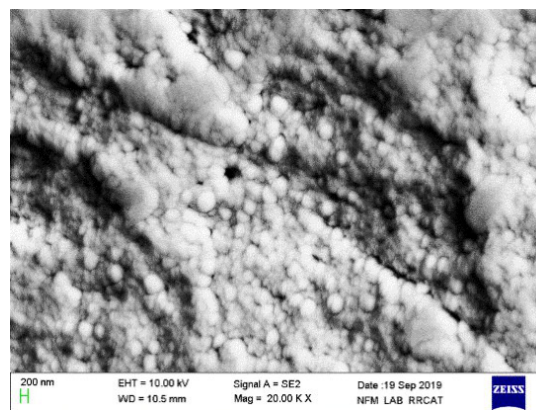


Fig. T.3.15: Morphology of the deposited NEG film on the aluminium substrate.

5.2.2 Chemical composition

NEG coated aluminium sample was examined by energy dispersive X-ray spectroscopy (EDXS) for chemical composition analysis. The chemical composition of deposited NEG film is Ti-28%, Zr- 32% and V-41% (atomic %). This chemical composition falls well within the specified range of reported quality-chemical composition map of Ti-Zr-V thin films suitable for low temperature (typical 180 °C x 24 hrs.) activation [13]. Attainment of this typical chemical composition confirms deployability of indigenously developed NEG coating in aluminium UHV chambers for our accelerator application.

6. Deployment, in-situ activation and experience with NEG coated beam chambers in Indus-2

Towards the end of year 2014, two Ti-Zr-V NEG coated beam chambers were deployed in Indus-2 for U1 & U2 undulators. Subsequently, one more NEG coated beam chamber for U3 undulator was augmented in the ring towards the end of year 2015. Total length of all the three NEG coated chambers is ~ 7900 mm with internal aperture of 17 mm x 81 mm. These chambers were coated by foreign industry as per RRCAT specification. On arrival, their vacuum performance was verified by activation and then successfully deployed in the ring, where they were re-activated in-situ [14]. Since their deployment all the three chambers are performing well and dynamic vacuum of $< 1 \times 10^{-12}$ mbar/mA with beam is stably maintained in these chambers without need of re-activation so far. Vacuum level in these chambers are better as compared to uncoated chambers in the ring. Photograph of U1 undulator with NEG coated beam chamber installed in Indus-2 is shown in Figure T.3.16.



Fig. T.3.16: U1 undulator with NEG coated chamber.

7. Conclusion

The present article summarised an overview of the R & D activities carried out, during recent years, on low activation temperature Ti-Zr-V NEG thin film compatible for accelerator UHV application and indigenous development of NEG coated aluminium alloy chamber for Indus-2 undulator. The activities involved indigenous design and development of various sub-systems of DC magnetron sputtering deposition system like: vacuum system, gas dosing system, solenoid & its power supply, bake-out & activation controller, data acquisition and control system and ultimate vacuum performance test setup.

The developmental activities also included designing suitable cathode as per chamber geometry requirement, synthesis of NEG films, characterisation, analysis of results, and optimization of process for the optimal NEG characteristics. Vacuum performance of NEG films deposited on SS and aluminium chambers have yielded highly encouraging vacuum performance results comparable with reported results by other labs. Measured data of pumping speed for H₂ & CO for the grown films is very useful for designing the vacuum systems for accelerator requirement. Spare undulator chamber for Indus-2 has been successfully coated with low activation temperature NEG film and stable desired static vacuum performance has been achieved. In view of large number of NEG thin film coated chambers requirement for upcoming 4th generation synchrotron radiation source, further works like: assessment of beam impedance of NEG coating thickness by simulation and measurement, dynamic vacuum performance in presence of synchrotron beam on Indus-2 beamline and evaluation of ageing consequent to activation-air venting cycles needs to be pursued. Deployment, in-situ activation and successful working of NEG film coated UHV chambers in Indus-2 for last seven years sufficiently demonstrates the capability of NEG thin film in line with expectation and has boosted our confidence for low conductance vacuum chambers in large numbers for upcoming 4th generation synchrotron radiation source. Indigenous technology of NEG film coated UHV chambers is aimed towards our contribution for Make in India initiative as part of AtmNirbhar Bharat mission.

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