Chapter 18

Development of Sealed off Vacuum Systems

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Most of the experiments in physics required vacuum environment. Research and development work in the field of thin film, semiconductor manufacturing, solar cell manufacturing, particle physics, plasma physics, fusion devices and accelerators required vacuum of the order of different scales. Creation of vacuum means extraction of gas molecules from the volume of the vacuum chamber. Sealed vacuum systems have many advantages over dynamic pumping vacuum systems. A sealed vacuum system is free from bulky primary and secondary pumps, so it is compact, lightweight, does not need electricity for maintenance of vacuum. As vacuum persists, as when required work can be done on the vacuum system. For field applications sealed vacuum systems are advantageous than dynamic pumping vacuum systems; as this can be easily transportable.

18.1. Gas Load

The existence of gas molecules inside the vacuum chamber is attributed to many factors. The gas load inside a vacuum chamber consists of two main categories i.e. external gas and internal gas. External gas consists of the atmospheric gases. The atmospheric gases like nitrogen, oxygen, argon, water vapor, helium, neon can penetrate either through leaks in the joints and in the chamber wall or by permeation through the chamber wall. The second category of the gas load is internal gas consists of gases due to outgassing, evaporation. The total gas load is equated as follows.

$$Q_G = Q_L + Q_{Per} + Q_D + Q_E \tag{18.1}$$

Q_L is due to gas load from leaks.

Q_{per} is due to permeation through materials like elastomers.

Q_D is due to outgassing.

Q_E is due to evaporation.

The order of vacuum inside a chamber is determined by the factor Q_G. The order of vacuum is inversely proportional to the total gas load Q_G. To reduce the value of Q_G, the mountable and demountable joints should be leak prove. To achieve a vacuum in the regime of ultra-high vacuum i. e. $<10^{-7}$ mbar, gross leak Q_L of the order of $< 10^{-9}$ mbar. L/sec is desirable. The leak may be of two types; real leak and virtual leak. The real leak rate $< 10^{-9}$ mbar.L/sec could be achieved by using proper sealing materials and perfect welding technique. Virtual leak rate could be minimized by welding from inside of the vacuum chamber. Apart from leak major contribution of the gas load comes from the outgassing. Outgassing is a process in which the adsorbed gas molecules are desorbed from the inside surface of the vacuum chamber wall. This property depends on the nature of the material and surface finish of it. To reduce the outgassing rate, the vacuum chamber and its components should be made of materials which are having low specific outgassing rate. The specific outgassing rate of materials used regularly in vacuum systems is given in the table 18.1. The data is for materials after baked out at 120 ^{0}C for 24 hours.

Table 18.1. Specific outgassing rate of UHV compatible materials.

Material	**Specific out gassing rate (Torr. L/sec.cm^2)**
SS304L	3 x 10^{-12}
Aluminium (6000 series alloys)	5 x 10^{-13}
Tungsten	1.6 x 10^{-10}
PEEK	6.2 x 10^{-12}
Alumina Ceramic	1 x 10^{-9}

OFHC Copper	1×10^{-12}
Glass	5×10^{-10}
Titanium	2×10^{-12}
Graphite	1.5×10^{-10}

Gas load due to evaporation contributes only when there is heating requirement inside the vacuum system otherwise this term can be neglected.

When a chamber is evacuated from atmosphere to ultra high vacuum regime, different gas dominates in different vacuum regime. The dominant gas in respective vacuum regime is given in the table 18.2.

Table 18.2. Gas species in different vacuum regime.

Pressure values (mbar)	**Gas species**
1000 (Atmosphere)	Air (N_2, O_2, H_2O, Ar, CO_2)
10^{-3}	H_2O (75% -95%)
10^{-6}	H_2O, CO
10^{-9}	CO, H_2
10^{-10}	CO, H_2
10^{-11}	H_2

During the evacuation of gases from the vacuum chamber there is a shift of actions from volume effect to surface effect. In the rough vacuum range the dominant gases are due to the atmospheric gases those are present in the volume of the vacuum chamber. Beyond medium vacuum range the dominant gases come from the surface of the chamber wall i.e. due to outgassing. Outgassing of the adsorbed gases on the surface of the chamber wall depends upon the desorption energy of the particular gas species. According to the desorption energy, there are three types of adsorbed gases; one with low desorption energy (< 19 kcal/mole), medium desorption energy (19-23 kcal/mole) and high desorption energy (> 23 kcal/mole). The outgassing effect arises from the adsorbed gases with low and medium desorption energy out of which the medium desorption energy creates more problem. The gases with low desorption energy comes out from the surface within few tens of minutes after initiation of the pumping but the gases with medium desorption energy emits slowly from the surface thus keeps the vacuum value low. The gases with high desorption energy seldom contribute to the outgassing rate. Desorption energy of water is 19-23 kcal/mole in stainless steel [1]. That is why the water vapor is dominant gas in the high vacuum regime as shown in the table 18.2. The pump down time for different gases with different desorption energy is shown in the Figure 18.1 [1].

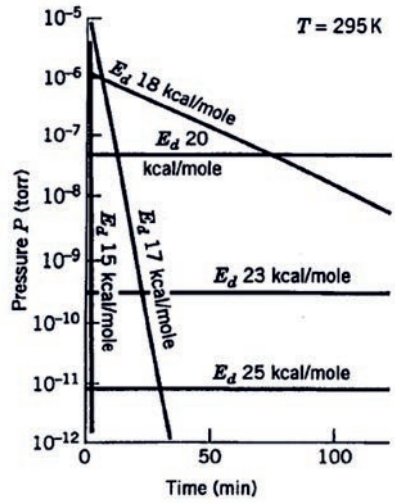

Figure 18.1. The pump down time for different gases with different desorption energy.

18.2. Requirement of Baking

To achieve vacuum of the order of $< 10^{-9}$ mbar, it is required to remove water vapor from the vacuum chamber. As the desorption energy of the water vapor is in the medium range, it is easy to remove it by baking the vacuum chamber. Depending on the nature of the material and sealing, baking can be performed at a temperature of 100 ^{0}C to 500 ^{0}C for 10-20 hours under vacuum. Higher temperature baking takes less time whereas lower temperature baking takes more time to remove the water vapor. Generally baking at 150 ^{0}C is sufficient for most of the cases but baking at 500 ^{0}C could remove chemisorbed water vapor as well as CO, CO_2, CH_4 etc. [2]. During baking, gases desorbed from the surface are evacuated from the chamber by the vacuum pump.

18.3. Selection of Vacuum Pumps

For development of the sealed off vacuum systems, baking is mandatory so that the gas load will be reduced. There are few vacuum pumps those works in ultra high vacuum regime like turbo molecular pump, sputter ion pump, cryo pump and non evaporable getter pump. In case of capture pumps like sputter ion pump, cryo pump, non evaporable getter pump, pumped gases stay in the pump body inside the vacuum chamber, hence the pump saturates in short span of time. In case of turbo molecular pump, the pumped gases exerted directly to atmosphere via rotary pump. So turbo molecular pump is suitable wherever baking is required. Turbo molecular pumps with different pumping speed from 10 L/sec to ~3000 L/sec are commercially available. The selection of proper pumping speed S (L/sec) of turbo molecular pump depends up on the through put Q (mbar. L/sec) and desired pressure P (mbar) according to the following relation [3].

$$S = \frac{Q}{P} \tag{18.2}$$

Throughput Q is the total gas load and can be estimated as per Eq. (18.1).

Advantages of sealed of systems are compactness, light weight, transportable etc. To satisfy these criteria, non evaporable getter pumps are suitable pumps for use in sealed of vacuum system as in situ pump. Non evaporable pumps keep on pumping gases inside a vacuum chamber without electric power. Non evaporable pump captures residual gases like Hydrogen, nitrogen, oxygen, carbon monoxide, carbon dioxide but do not pump noble gases like helium, neon and argon. Non evaporable pumps are available in different sizes with different pumping speed. Commercially available non evaporable getter pumps ranges from 50 L/sec to 3500 L/s pumping speed for hydrogen.

18.4. Experimental Set Up

The experimental set up is shown in the Figure 18.2. It consists of a main vacuum chamber C1. It is pumped by one turbo molecular pump of pumping speed 80 L/sec and backed by one rotary pump connected in series. One 63 CF gate vale V1 is used to isolate the main vacuum chamber from the turbo molecular pump. G1 is a hot cathode ionization gauge to measure the total pressure of the chamber. RGA is a residual gas analyzer [4] used to measure partial pressure of residual gases. C2 is the chamber to be sealed is attached in series with the chamber C1 by one 35 CF right angle valve V2. G2 is a full range vacuum gauge and N1 is a non evaporable getter pump. Depending upon the size and materials of the chamber C2, the size of N1 can be decided. V3 is a fine tuned gas leak valve used for injection of process gas into the chamber wherever required.

The procedure for the sealing is described briefly elsewhere [5]. After assembling all the embodiments, the whole system was leak tested by using one helium leak detector. Leak rate of the order of $< 10^{-9}$ mbar. L/sec was achieved. Vacuum up to 10^{-2} mbar was created by rotary vacuum pump and then turbomolecular pump was switched on. When the vacuum of the order of 10^{-7} mbar was achieved baking of the whole system was carried out. For baking, silicon insulated heating tapes were wrapped uniformly throughout the vacuum chamber outer wall. The heating tapes were powered by one Variac. Temperature was monitor by K-type thermocouple and the temperature of the vacuum chamber was controlled by the Variac. The system was baked at a temperature up to 150 ^{0}C for 24 hours.

During baking the non evaporable getter N1 was kept at condition mode i.e. at a temperature of 150 ^{0}C – 200 ^{0}C followed by activation for one hour at a temperature of 500 ^{0}C at the end of the baking cycle. After baking the system was allowed to cool down to room temperature and total pressure of the order of 10^{-9} mbar was achieved. Before baking the partial pressure of the water vapor that was measured by residual gas analyzer was of the order of 10^{-7} mbar whereas after baking it came down to the order of 10^{-9} mbar. At this point of time the valve V2 was closed and the chamber C2 was isolated from the main vacuum chamber.

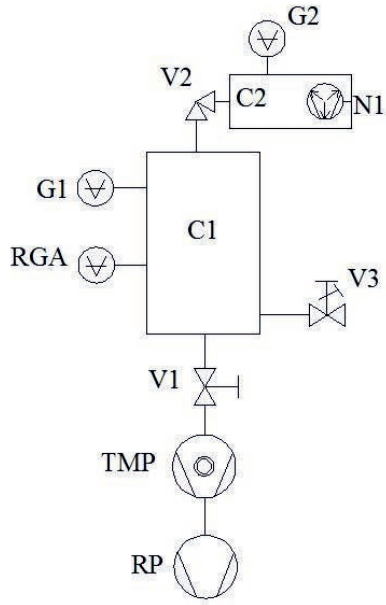

Figure 18.2. Schematic diagram of the experimental set up. RP- Rotary Pump, TMP- Turbo Molecular Pump (80 L/sec pumping speed), V1- 63 CF Gate Valve, RGA- Residual Gas Analyzer, G1- Hot Cathode Ionization Gauge, V2 – 35 CF Right Angle Valve, G2 – Full Range Vacuum Gauge (PKR 251), N- Non Evaporable Getter Pump, C1 – Main Vacuum Chamber, C2- Sealed off Vacuum Chamber, V3- Fine Tuned Gas Leak Valve.

18.5. Results

Three types of sealed off vacuum systems are developed in our laboratory. Those are sealed Flash X-ray (FXR), sealed backward wave oscillator (BWO) and sealed miniature dense plasma focus (DPF) device. Depending on applications materials were selected and same procedure for sealing was followed as describe above.

18.5.1. Sealed off FXR

Flash X-ray device generates pulsed x-ray with nano second pulse width this is a cold cathode consisting of a ring shaped cathode of 10 mm inner diameter made of SS and a tapered anode of 1mm diameter made of tungsten. The anode and cathode assembly is housed in an SS 304 L tube. A vacuum interface of Aluminum disc is used as X-ray window. One feed through made of natural PEEK is used as high voltage insulation between the tube wall and the anode. As the dimensions of the vacuum tube are non standard and requirement for PEEK to metal seal; vacuum sealing of indium wire is preferred. One 50 L/s non evaporable getter (NEG) is used for in situ pumping of the FXR tube in seal off condition. The schematic diagram of the FXR tube is shown in the Figure 18.3 [6]. In this case baking was carried out at a temperature of 110 ^{0}C for 24 hours.

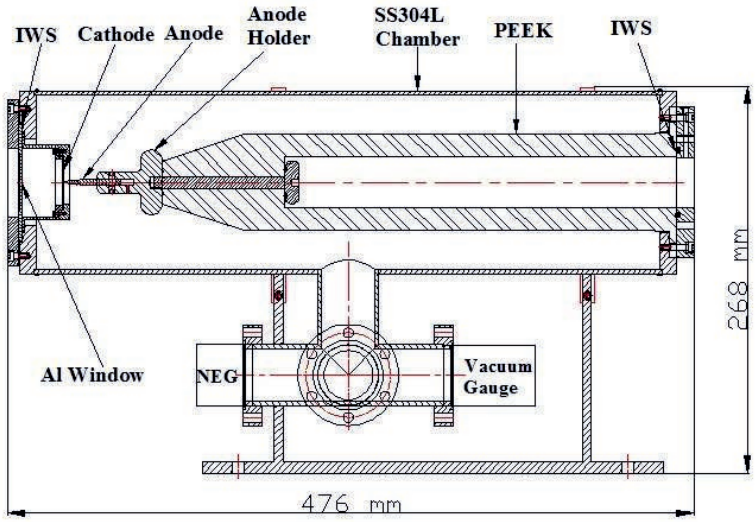

Figure 18.3. Schematic diagram of the FXR tube. Dimensions are in mm.

After sealing, vacuum of the order of 5 x 10^{-7} mbar was maintained. It was operated for x-ray generation. During X-ray generation, vacuum in the tube was raised up to a vacuum of the order of 10^{-5} mbar and was pumped by the NEG to the base pressure. Evolution of gases during X-ray generation and operation parameters are shown in the table 18.3.

Table 18.3. Operational Parameters of FXR tube.

Shot. No.	**Interval between shots (minutes)**	**Base pressure (mbar)**	**Operating voltage (kV)**	**X-ray dose at 50 cm from X-ray window (mSv)**
1	0	5.8 x 10^{-7}	315	0.3
2	2	5.0 x 10^{-7}	315	0.3
3	2	5.0 x 10^{-7}	315	0.3
4	2	5.1 x 10^{-7}	315	0.3
5	2	5.3 x 10^{-7}	315	0.3

18.5.2. Sealed off BWO

Backward wave oscillator is a high power microwave generator [7]. The materials used in this system were selected based on their better outgassing rate. The vacuum chamber of BWO consists of stainless steel 304L for chamber wall and flanges, alumina ceramics for high voltage insulation, quartz window for transmission of microwave and graphite for the electron emission. Standard CF Metal gaskets were used in the demountable joints. All the embodiments were assembled and helium leak testing was performed. A gross leak rate of the order of 5 x 10^{-11}

mbar L/sec was achieved. Initial pumping of the chamber was carried out by turbo molecular pumping station followed by baking at a temperature of 150 ^{0}C for 20 hours. At the end of the baking cycle the NEG was activated for one hour. When the chamber was cooled down to room temperature the BWO vacuum chamber was isolated from the TMP station and a vacuum of the order of 3 x 10^{-8} mbar was maintained. The schematic diagram of the BWO vacuum chamber is shown in the Figure 18.4.

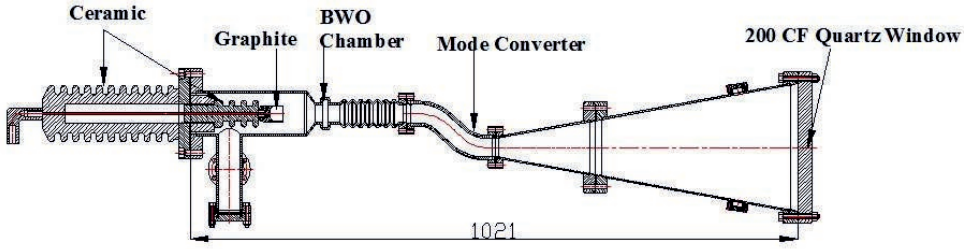

Figure 18.4. Schematic diagram of the BWO vacuum system. Dimension is in mm

18.5.3. Sealed off miniature DPF

Dense plasma focus device is used as a fusion device [8, 9]. Fusion takes place between deuterium-deuterium or deuterium-tritium due to the pinching effect of the electromagnetic field. Pulsed electric current passes through the gas between two anode and cathode and ionize the gas into plasma. This plasma pinch down on itself at the end of central cylinder to increase density and temperature of the plasma. Particles like electrons, ions and x-ray are generated from the pinch along with neutrons. One such device was developed in our laboratory. Sealed of DPF is different than sealed FXR and sealed BWO vacuum chamber. In sealed DPF it is required to maintain vacuum as well as supply deuterium gas for operation. Non evaporable getter is used as source of deuterium [5].

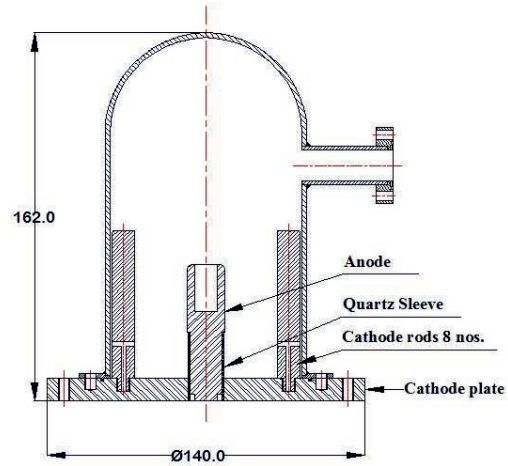

Figure 18.5. Schematic diagram of miniature dense plasma focus. Dimensions are in mm.

Figure 18.5 represents the schematic diagram of a miniature dense plasma focus. Indium wire seal is used as seal between vacuum chamber and the cathode plate. One non evaporable getter pump with pumping speed of 20 L/sec for hydrogen is used for in situ pumping and as a deuterium replenisher. The same procedure was followed like sealed FXR e. g. initial pumping by TMP, baking at 110 ^{0}C for 24 hours but after cooled down to room temperature the NEG was charged with deuterium gas [5] and sealed and isolated from the main vacuum system. In sealed condition it was operated for neutron generation at ~9.5 kV charging voltage and ~180 kA discharge current at a filled deuterium pressure of 8 mbar. Average neutron yield of ~10^7 n/pulse was observed by silver activation counter for 15 consecutive shots.

18.6. Conclusion

In conclusion, three different types of devices were sealed in ultra high vacuum range. FXR was operated for x-ray generation and vacuum stability. Sealed miniature DPF was tested for neutron generation. BWO vacuum chamber was sealed and will be operated in near future.

References

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