Removal of water from unbaked vacuum system

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Abstract: The water vapour contained in the atmosphere contaminates a vacuum systems, when it is exposed to air. The degree of contamination will depend on the surface characteristics, treatment of the system walls, the relative humidity and the turbulence of air, the amount of time the system is opened to the atmosphere, etc. If the temperature of the exposed surface is low, possibility of water condensing on the surface exists. In order to study the effect and optimise procedure for achieving high vacuum in a vacuum chamber initially containing water, a vacuum chamber has been fabricated. Pump down time has been measured for different amount of water placed in the vacuum chamber with varying pumping speeds. The paper presents a review of similar activities and results of the experimental study.

1. Introduction:

The beam chamber of VEC superconducting cyclotron [1] has a number of penetrations in the median plane in between the upper and lower superconducting coils to accommodate extraction components and beam diagnostic components. Because of the compact structure, adequate space was not available at the penetrations for placing a thermal shield. The temperature at these penetrations is quite low. It is observed that moisture from air condenses there and considerable time is taken to achieve good vacuum, specially, in monsoon. In the case of water leakage in the system, considerable amount of water gets accumulated in the beam chamber. Because of the complex geometry of the cyclotron, it is quite difficult to remove water from the restricted areas. It is also not possible to bake the acceleration chamber to enhance the rate of moisture removal.

An experimental set-up has been fabricated to study the pump down characteristics in the presence of water in a vacuum enclosure. The paper presents a literature survey on work carried out on similar lines and results of experiments carried out.

2. Review of similar activities

Chan 2010 [2] examined the effect of environment humidity on the rate of thermal outgassing from an aluminium chamber. An aluminium glove box and an air shower, which provided dehumidified environments with water vapor concentrations of 0.1 ppm and 5 ppm respectively, were utilized to assess the effect of environmental humidity on the rate of thermal outgassing. Chan observed that only 4 h of evacuation was required to yield a small rate of the order of 1 x 10^{-13} mbar L s^{-1} cm^2 of thermal outgassing upon filling with super-dry nitrogen without baking. A smaller rate of thermal outgassing was obtained using a drier venting gas, or by exposure to a drier environment. A thermal outgassing rate of the order of 1 x 10^{-11} mbar L s^{-1} cm^2 was achieved by venting the glove box with super-dry nitrogen inside and controlling of environmental humidity at 0.1 ppm.

Yamazaki 2010 [3] carried out control of water vapor in a nitrogen gas purge line in addition to surface treatments of chambers using buff polishing and electrolytic polishing, followed by measurement of outgassing rate of the chambers. He observed that with proper surface treatment and introduction of well controlled nitrogen gas to the vacuum system which was not baked out reduces the pump down time considerably.

Liapis 2008 [4] constructed expressions for determining the exergy inputs and exergy losses due to heat and mass transfer in the primary and secondary drying stages as well as in the water vapor condenser and vacuum pump of the process involving freeze drying of pharmaceuticals in vials on trays. The exergy expressions can be used for operational control policies to minimize the irreversibilities occurring in the operations of a freeze drying system of a given design, thus enhancing the efficiency of energy utilization.

Chen 2007 [5] studied the morphological changes of water in a vacuum cooling system in the course of vacuum chamber pressure step down. The experiment result shows that steam adhered to vacuum chamber due to the atmospheric pressure evaporates as the chamber pressure decreases and produces a great amount of smoke after the vacuum pumping system is initiated for 20 s. After 115.2s, the liquid water in the vessel begins to produce tiny bubbles and then large bubbles at 121.2 s, and boils at 126.7 s. After the rapid boiling is over, the liquid water enters the freezing stage at 181.5 s. When the vacuum pumping system is stopped at 480 s, the liquid water inside the vessel is frozen into solid ice which is consisted of two layers, irregular porous layer on the top and dense layer on the bottom. The cooling rate of liquid water in this experiment is between 0.33–0.38 ^{0}C/s, and 0.04–0.07 ^{0}C/s during the freezing process.

Caleman 2006 [6] studied the evaporation of pure water clusters under vacuum. He simulated the evaporation of water molecules. The average number of evaporated water molecules per surface area is 0.9–1.0 molecule/nm^2. The evaporation becomes slow, once the temperature reaches 240 K. We find that as the evaporation ceases, the temperature falls to about 215 K. He observed that the cooling rates are in good agreement with experimental results, and evaporation rates agree well with a phenomenological expression based on experimental observations.

Dostal 2004 [7] has developed a simple mathematical model of the vacuum cooling process which enables to predict a temperature evolution regarding an equipment size, vacuum pump parameters and properties of the cooled liquid. Real thermophysical properties of the cooled liquid were considered in the model along with the assumption that the main resistance against mass transport is situated on the side of liquid phase. Parameter identification of mass and heat transfer coefficients were based on available experimental data.

Song 2002 [8] developed a numerical code to predict vacuum freeze drying processes in trays and vials using a finite volume method to discretize the governing partial differential equations. Along with the finite volume method, a moving grid system was adopted to handle irregular and continuously changing physical domains encountered during the primary drying stage. To show the validity of the present calculation scheme, freeze drying in a tray was simulated and the results were compared with available experimental data.

Akaishi 1997 [9] experimentally investigated the effect of helium glow discharge cleaning as a wall conditioning technique for the production of ultrahigh vacuum in an unbaked vacuum system. A test chamber made of 304 stainless steel was constructed. The chamber was initially exposure to air before pump-down. Discharge cleaning was done at the constant helium ion fluence of 0.5 Coulomb/cm^2. It was observed that the pumping time to attain the pressure of order of magnitude of 10^{-8} Torr is shortened after the discharge cleaning by one fourth compared with the pumping time without discharge cleaning. But the ultimate pressure at the pumping time of 72 h after the discharge cleaning is rather the same as that without discharge cleaning.

Porta 1996 [10] has recommended the use of getters for removal water in sealed devices for semiconductor processing, vacuum insulated vessels and other applications

Berman 1995 [11] examined the problem associated with water vapour in vacuum system and methods to remove it. In order to reduce the desorption rate and remove large amounts of water vapour Berman suggests the use of methods for conditioning the construction materials and transfer energy to the adsorbed gases to break their bonds. Berman observes that traditional vacuum bakeout cannot be used with some applications such as accelerators, or fusion devices. Other methods such as flushing with cold or hot neutral gas, glow discharge, or either electron, ion, or ultraviolet radiations have been successfully used.

Novikov 1968 [12] examined the mechanism of vacuum sublimation of ice with experimental data and theoretical results. He observed that in the vacuum chamber very favourable conditions are created for external mass transfer, when small fluctuations of pressure can cause mass transfer velocities of several meters per second. In the presence of an intense source of mass release (sublimation of ice in a vacuum) and in the absence of leakage the total pressure in the chamber may be assumed equal to the saturation pressure and the chamber may be considered to contain vapor only. In this case, the rate of mass transfer is determined by the vapor pressure drop.

3. Experimental set-up

A vacuum system (Fig. 1) has been set up to study removal of water from the enclosure and optimisation of the procedure for removal of water and subsequent achievement of high vacuum in it. The vacuum chamber is connected to a scroll pump with pumping speed 7 l/sec through a 25 mm port. A turbo molecular pump with pumping capacity 210 l/sec is also connected to the vacuum system using another 25 mm port. The effective pumping speed at the vacuum chamber for the scroll and turbo molecular pump was evaluated and found to be about 0.7 l/sec and 0.6 l/s respectively. Vacuum gauges G1 and G2 are placed close to the vacuum chamber and the turbo molecular pump respectively. An extension having a glass view port has been added to visualise removal of water from a glass beaker partially filled with water. The total volume of the chamber is about 7 litres. Pneumatic valves GV1 and GV2 and used to isolate scroll pump and turbo molecular pump respectively.

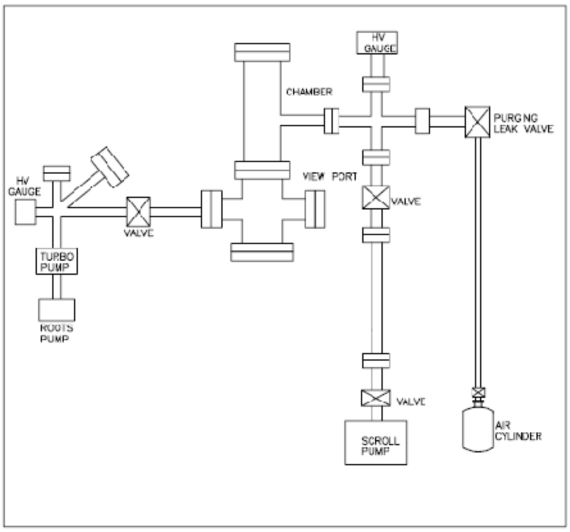

Fig. 1: Schematic diagram of vacuum system

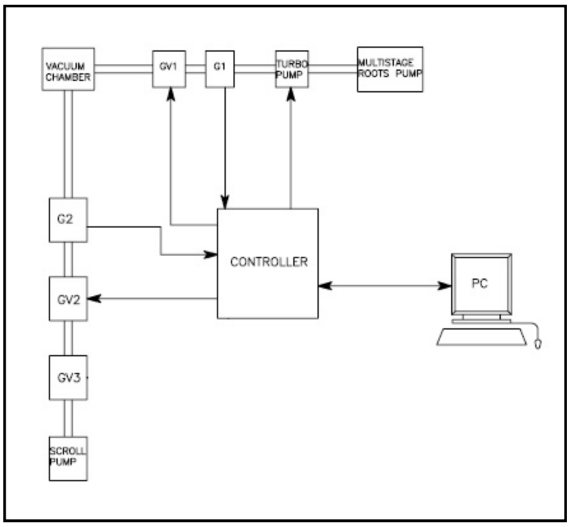

Fig. 2: Control diagram for vacuum system

A control circuit (Fig. 2) is used to sequentially operate the valves, backing and high vacuum pump. Two compact gauges monitor the vacuum in the system. The valves and vacuum pumps are operated using the relays available in the gauge controller. The scroll pump is initially operated till the vacuum in the vacuum reaches 2×10^{-2} mbar. The high vacuum pump is then connected to the vacuum system. The gauges are connected to a computer for data acquisition. Safety interlocks have also been incorporated to isolate the turbo molecular pump in case of power failure and accidental pressure rise in the vacuum chamber.

4. Results

The different stages of pump down was observed through the view glass. A webcam was placed in front of the view glass and the evacuation of the chamber containing a glass beaker of water was observed. It was observed that as pumping progresses vigorous boiling of water is observed. After some time, the boiling phenomena stops, the water solidifies and ice is formed. The amount of ice reduces slowly. Pressure data in the vacuum chamber is stored on-line in a computer. The quantity of water was varied from 2.5 gram to 20 gram. Fig. 3 shows the variation of pressure with time. In order to assess the effect of exposed surface, the experiment was carried out by varying the exposed area from 11 sq. cm to 50.9 sq. cm (Fig. 4). The effect of speed of pumping was evaluated by changing the conductance of the scroll pump connected to the vacuum system. The variation of pressure with time for orifice diameters 2.5mm to 9 mm is given in Fig. 5. The orifice diameter is chosen to reduce the conductance by 25%, 50% and 75% respectively. Fig. 6 shows the effect of pumping with the scroll pump.

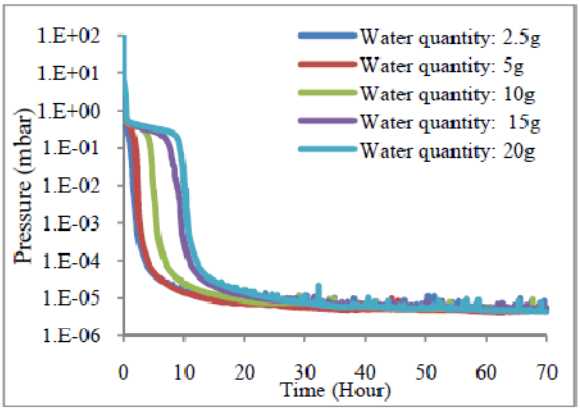

Fig. 3: Pump down-different mass

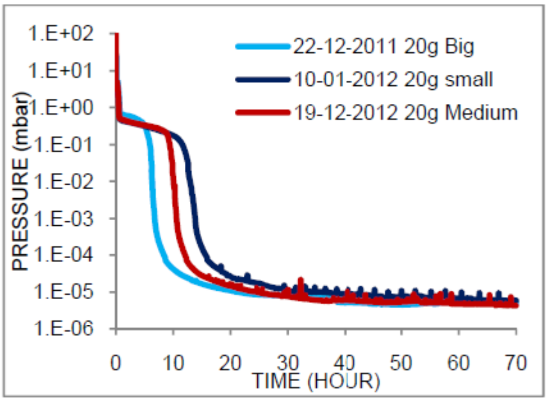

Fig. 4: Pump down-different area

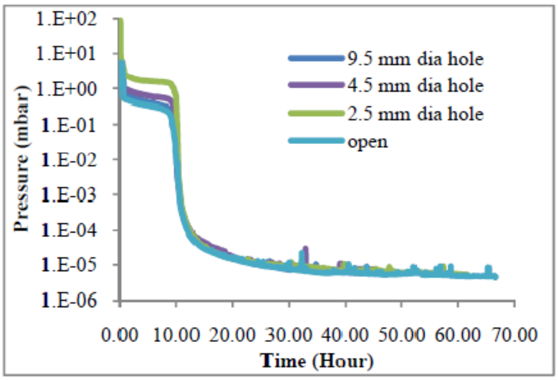

Fig. 5: Pump down-different conductance

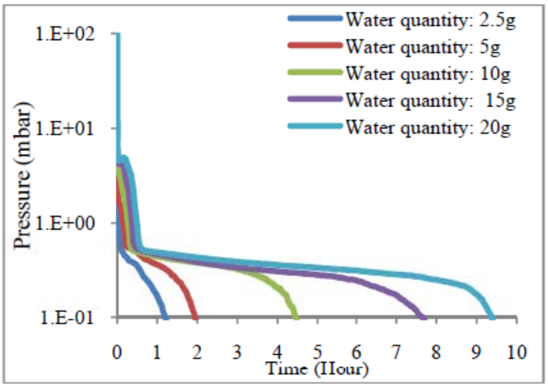

Fig. 6: Pump down – initial period

It was observed from the experiments (Fig. 3) that after the end of 70 hours of pumping, the pressure in the system was about 5.52×10^{-6} mbar, 4.47×10^{-6} mbar, 4.64×10^{-6} mbar, 5.1×10^{-6} mbar and 4.32×10^{-6} mbar for initial water of 2.5 g, 5 g, 10 g, 15 g and 20 g water respectively. The initial rate of water removal is highly dependent on the surface area, but the effect after about 70 hours is not so prominent (Fig. 4). The pressure in the system was about 7.3×10^{-6} mbar, 6.54×10^{-6} mbar, 5.96×10^{-6} mbar and 4.32×10^{-6} mbar respectively for initial water of 20 g water after the end of 70 hours of pumping, when the conductance was 25%, 50%, 75 % and 100%. The time taken to achieve a vacuum of 0.5 bar was about 8 min, 30 min, 30 min, 42 min and 50 min for initial water of 2.5 g, 5 g, 10 g, 15 g and 20 g water respectively (Fig. 6).

5. Discussion

Removal of water using vacuum has been studied for a long time. It has application in electronic industry [10], food processing [8,13,14], food preservation [5, 7], desalination [15], pharmaceutical industry [16], accelerators [17], tokomaks [18], etc. Different forms of heating, viz., microwave, thermal, dry gas purging, glow discharge, etc. have enhanced removal of water.

The literature survey and experiment have shown that during the evacuation process, initially free water evaporates at the surface of water. As the pressure reduces, the boiling point of water reduces and boiling of water is observed. Boiling continues and the liquid transforms to vapours. The corresponding amount of heat, the latent heat, is continuously supplied from the system. The value of latent heat is rather large (order of MJ/kg) and it play a substantial role in the energy balance of liquid phase. The balance includes the flow of water vapours which leave the liquid surface and which actually contain the energy of the latent heat. This energy is, therefore, taken off the liquid phase and in the absence of a heat input the liquid temperature decreases. The temperature of the vessel is high. So, different modes of boiling can be observed. The lower temperature would reduce the partial pressure of vapours below the total pressure and the boiling process would terminate. Once, water converts to ice, removal of water is by sublimation.

6. Conclusions

It was observed from the experiments that the long term performance of the vacuum is not highly dependent on the initial amount of water. The rate of removal of water is dependent on the surface area and conductance.

It is further studies have to be continued. It is planned to add a residual gas analyser to the vacuum system and assess the effect of residual amount of H_2O after about 70 hours for different amount of initial water. A vacuum gauge for accurately measuring the pressure from 1 bar to about 0.01 bar is also planned to minutely observe the initial pressure change.

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