Study of Hydrogen Pumping through Condensed Argon in Cryogenic pump

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Abstract. In ultra high vacuum (UHV) range, hydrogen is a dominant residual gas in vacuum chamber. Hydrogen, being light gas, pumping of hydrogen in this vacuum range is limited with widely used UHV pumps, viz. turbo molecular pump and cryogenic pump. Pre condensed argon layers in cryogenic pump create porous structure on the surface of the pump, which traps hydrogen gas at a temperature less than 20°C K. Additional argon gas injection in the cryogenic pump, at lowest temperature, generates multiple layers of condensed argon as a porous frost with 10 to 100 Å diameters pores, which increase the pumping capacity of hydrogen gas. This pumping mechanism of hydrogen is more effective, to pump more hydrogen gas in UHV range applicable in accelerator, space simulation etc. and where hydrogen is used as fuel gas like tokamak. For this experiment, the cryogenic pump with a closed loop refrigerator using helium gas is used to produce the minimum cryogenic temperature as ~ 14°C K. In this paper, effect of cryosorption of hydrogen is presented with different levels of argon gas and hydrogen gas in cryogenic pump chamber.

1. Introduction

In cryogenic technology, pumping through cryosorption is widely used, to achieve high pumping speed and capacity of non condensable gases (H₂, D₂, He, Ne). Cryosorption also provides the highly cleaned vacuum condition to achieve extreme vacuum. The adsorbents like active carbon (charcoal), zeolite molecular sieves are mostly used for cryosorption pumping in cryogenic pumps, i.e. Refrigerator cooled cryopump, Bath cryopump, Supercritical helium cooled cryopump. These adsorbent materials have large surface area to capture non condensable gases below 20°C K temperature. But all cryogenic pumps are designed with limited adsorbent material for particular application. Thus these adsorbents have limited capacity to pump non condensable gases at saturation level. To improve this pumping limitation, A Cryosorption by condensed gases like Ar, CO₂, SF₆ below 20°C K temperature also used as creative adsorbents to increase pumping speed and capacity of non condensable gases. Argon gas is more preferable for condensed gas adsorbent than other gases, CO₂, SF₆. As an adsorbent, Argon has effective properties for cryosorption, like high thermal conductivity; non reactive, inert gas etc. There are two types of gas filling of adsorbent gases in cryopump, as pre condensed (frost) and continuous condensed. In pre condensed [1], adsorbent gas is filled sufficiently in cryopump to create porous structure before actual pumping of main vacuum chamber. It is, useful to achieve extreme vacuum condition and option of saturated static adsorbents (activated charcoal, molecular sieves) to increase pumping capacity and speed. Gradually, the pumping efficiency is reduced with time in pre condensed technique due to filling of pores with fresh gas load. In continuous
condensed [2], adsorbed gas is filled continuously in cryopump with actual pumping of the vacuum chamber to get effective pumping speed of non condensable gases. Continuous condensed of adsorbent gas is used to determine cryosorption pumping effect on non condensable gases and also provides continuous pumping of these gases with fresh frost.

ADITYA [3] is ohmically heated limiter based, moderate size Tokamak. The ADITYA vacuum vessel is pumped through three turbo molecular pumps and a refrigerator cooled cryopump. Hydrogen is the fuel gas to produce various discharges like high temperature plasma discharge, glow discharge cleaning (GDC) [4] etc. For wall conditioning, Glow discharge cleaning is operated with high pressure of hydrogen and for long duration for many hours. The resultant of GDC, hydrogen pressure is increased significantly in vacuum vessel, which perturbs the high temperature plasma discharge as recycling of hydrogen in hot plasma. Thus, the experimental study of cryosorption using argon adsorbent to increase pumping speed and capacity of hydrogen has been carried out to improve pumping efficiency of ADITYA vacuum vessel.

In this experiment, the refrigerated cooled cryopump has been used at cryogenic temperature 14ºK of 2nd stage as a manufactured parameter.

2. Experimental set-up
The schematic view of this experimental setup is described in Figure 1. The refrigerated cooled cryopump of 250 torr.liter crossover evacuates the SS304 vacuum chamber to achieve ultra high vacuum. Before cryopump operation, rotary pump is used to evacuate the cryopump and vacuum chamber simultaneously to get crossover pressure. For total and partial pressure measurement, Ionization gauge and residual gas analyzer are used. Two gas leak valves are used to fill gases as argon and hydrogen.

![Schematic view of the experiment setup](image)

Fig. 1. Schematic view of the experiment setup to study of the hydrogen pumping through condensed argon in cryogenic pump.

Other important parameters of this cryopump are: (1) pumping speed (liters/sec): 9500 for water vapor, 4000 for nitrogen, 3500 for argon, 6500 for hydrogen (2) crossover: 250 Torr liters for N₂ (3) capacity: condensable gases greater than 2200 standard liters at 5 x 10⁻⁶ torr and hydrogen 30 standard liters at 5 x 10⁻⁶ torr (4) maximum argon throughput at 20ºK: 11 torr.liter/sec (5) minimum temperature on 2nd stage: 14ºK
3. Theoretical Basis

In cryogenic pumping technology, Effectiveness of cryo pumping is significantly dependent on sticking co-efficient \( \alpha \), which is the ratio of number of particles stick on cryo surface to number of particles impinge on cryo surface. This ratio stays between zero (no pumping) to one (100 % pumping of impinging particles) for cryo surface. Sticking coefficient \( \alpha \) derives from equation (1) [5] for commercially available refrigerator cooled cryopump with two components (baffle and cryo panel) connected in parallel.

\[
\frac{1}{\alpha} = \frac{1}{c} - \frac{1}{w} + 1
\]  

(1)

Here, \( c \) is capture coefficient, which characterize the cryo pump’s pumping speed. Capture coefficient \( c \) is the ratio of actual pumping speed \( S \) to theoretical black hole pumping speed \( S_{id} \) and derives from equation (2) [6].

\[
S = c \cdot S_{id} = c \cdot A \left[ \frac{R_0 T}{2 \pi M} \right]^{1/2}
\]

(2)

Thus, the pumping speed of cryo pump depends on geometry as inlet area \( A \) and types of gas being pumped as mass \( M \). Here \( R_0 \) is gas constant and \( T \) is temperature as 273.15ºK [6]. The pumping speed \( S \) is also measured using general formula of throughput in vacuum system.

\[
S_{measured} = \frac{Q}{P}
\]

(3)

Throughput or gas influx \( Q \) is adjusted using gas flow controller and measured pressure of particular gas or total pressure for temperature 293ºK. In equation (1), \( w \) is transmission probability, which reduces the ideal pumping speed of cryopump, due to \( w \) depends on baffle designed to restrict gas particles movement to cryo panel. In this experiment, baffle design data of cryopump is not available. Thus, transmission probability is taken by Ref [6] as 0.25 for hydrogen pumping of standard refrigerator cryopump. The hydrogen sticking coefficient \( \alpha_{H_2} \) on condensed argon can be determined for 14ºK temperature of refrigerator cooled cryopump using above equations and the measured pumping speed of hydrogen with different gas load of argon.

4. Experimental results and Discussions

The cryosorption effect of continuous condensed argon on hydrogen at temperature 14ºK is shown in figure 2. Hydrogen is pre filled (before argon filling) in cryopump chamber with high quantity as more than 10 liters to achieve saturated condition of cryosorption of activated charcoal on 2nd stage of the cryopump. This affects hydrogen background level stable on 5 x 10⁻⁷ torr and this background pressure is enough to realize the pumping effect of continuous condensed argon. During continuously increasing gas feed of argon, when argon pressure is reached at 1 x 10⁻⁵ torr (point A in figure 2), the hydrogen pumping is started on enough argon frost structure. Gradually, the hydrogen pumping is increased with argon pressure increasing. When argon gas feed is stopped at point E, the hydrogen pressure is increased suddenly and stable on 1 x 10⁻⁷ torr, which is reduced in factor of 5 from the initial saturated pressure.
Fig. 2. Cryosorption effect of continuous condensed argon on saturated level of hydrogen, at 14°K temperature in cryopump. Various pressure levels of argon are shown as points A to E, where argon pressure at (A) $1 \times 10^{-5}$ torr (B) $5 \times 10^{-5}$ torr (C) $6 \times 10^{-5}$ torr (D) $8 \times 10^{-5}$ torr (E) Argon gas feed off.

The cryosorption effect of condensed argon on hydrogen during simultaneously filled argon and hydrogen in cryo chamber at temperature 14°K is shown in figure 3. In this experiment, continuous filled hydrogen throughput is steadied on pressure $1.3 \times 10^{-7}$ torr. Gradually increasing of argon pressure reaches at $1.3 \times 10^{-5}$ torr, steady filled hydrogen pressure starts to reduce.

Fig. 3. Cryosorption effect of argon adsorbent, the continuous increasing of argon, which effect on continuous filled hydrogen at $1.3 \times 10^{-7}$ torr with 14°K temperature

This effect indicates the cryosorption due to sufficiently filled argon creates condensed porous structure to trap hydrogen molecules. With argon pressure increasing in $10^{-5}$ torr range, hydrogen is reduced
linearly up to pressure $5 \times 10^{-8}$ torr. The argon filling is limited due to operation limit in $10^{-4}$ torr range of residual gas analyzer (RGA). Thus, we cannot distinguish limitation of hydrogen pumping with continuous increasing of argon at temperature $14^\circ$K.

The sticking coefficient is a universal parameter to measure cryo-pumping effect. Using experimental data of figure 3 and theoretical basis, the sticking coefficients of hydrogen ($\alpha_{H_2}$) are calculated for different levels of argon gas load on cryopump at temperature $14^\circ$K. In figure 4, the sticking coefficients of hydrogen ($\alpha_{H_2}$) are plotted with various argon gas loads in torr.liter/cm$^2$. The hydrogen sticking coefficient ($\alpha_{H_2}$) is calculated maximum as 0.34 on argon gas load $3.2 \times 10^{-6}$ torr.liter/cm$^2$ and minimum as 0.14 on argon gas load $1.4 \times 10^{-6}$ torr.liter/cm$^2$. The maximum value 0.34 of hydrogen sticking coefficient ($\alpha_{H_2}$) can be increased with argon gas load increasing. It is limited due to measurement limit of RGA in this experiment.

![Graph showing hydrogen sticking coefficient vs. argon gas load](image.png)

**Fig. 4.** The sticking coefficients of hydrogen ($\alpha_{H_2}$) with various argon gas loads in torr.liter/cm$^2$ at temperature $14^\circ$K through steady gas feed of hydrogen as $1.3 \times 10^{-7}$ torr.

### 4. Conclusion

The initial result of continuous condensed argon adsorbent effect on hydrogen at temperature $14^\circ$K of refrigerator cooled cryopump has been concluded. The cryosorption effect of argon frost on hydrogen is started on argon gas feed pressure near $1 \times 10^{-5}$ torr in this cryopump system. The sticking coefficient of hydrogen ($\alpha_{H_2}$) at temperature $14^\circ$K is determined to be 0.34 for continuous condensed argon on argon gas load $3.2 \times 10^{-6}$ torr. liter/cm$^2$ and steady gas feed of hydrogen at pressure $1.3 \times 10^{-7}$ torr. The final value of sticking coefficient of hydrogen ($\alpha_{H_2}$) at temperature $14^\circ$K in this cryopump can be determined with changing in experimental setup of measuring instruments. It is noticed that the second stage temperature of this cryopump is decreased up to $12^\circ$K from its standard manufactured value as $14^\circ$K after sufficiently filled argon (pre condensed effect). This effect is useful to increase pumping capacity and speed of non-condensable gases. These experiments more depend on cryo surface temperature, so experiments should be carried out at lower temperature of cryo pump. This initial study is motivated us to further experimental studies of pre condensed argon effect, increasing hydrogen pumping in actual vacuum vessel of ADITYA Tokamak.

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