

Experimental Investigation on Activated Carbon Based Cryosorption Pump

K. B. Vinay, K. G. Vismay, S. Kasturirengan, G. A. Vivek

Abstract—Cryosorption pumps are considered safe, quiet, and ultra-high vacuum production pumps which have their application from Semiconductor industries to ITER [International Thermonuclear Experimental Reactor] units. The principle of physisorption of gases over highly porous materials like activated charcoal at cryogenic temperatures (below -1500°C) is involved in determining the pumping speed of gases like Helium, Hydrogen, Argon, and Nitrogen. This paper aims at providing detailed overview of development of Cryosorption pump and characterization of different activated charcoal materials that optimizes the performance of the pump. Different grades of charcoal were tested in order to determine the pumping speed of the pump and were compared with commercially available Varian cryopanel. The results for bare panel, bare panel with adhesive, cryopanel with pellets, and cryopanel with granules were obtained and compared. The comparison showed that cryopanel adhered with small granules gave better pumping speeds than large sized pellets.

Keywords—Adhesive, cryopanel, granules, pellets.

I. INTRODUCTION

CRYOSORPTION pumps belong to the class of Entrapment/capture vacuum pumps wherein they retain the gas molecules by sorption and/or by condensation on the internal surfaces [1]. Now-a-days commercially available cryosorption pumps made especially for industrial application operate in the temperature range of 20-30K, since they do not require the pumping of lighter gas molecules like hydrogen, deuterium and helium etc. These pumps involve the use of sorption materials like activated charcoal which have optimum adsorption characteristics in the cryo-temperatures. Materials like Zeolite, silica gel and other sorption materials do not have prominent application in the area of cryosorption compared to activated carbon. Cryosorption pumps are very much required for the optimum plasma performance in International Thermonuclear Experimental Reactor [ITER]. The Cryosorption pumps will extract the helium ash generated by the deuterium-tritium fusion reaction, this application makes the cryosorption pumps prominent [2]. In this experiment, we are aiming at understanding the performance of cryosorption pump with the use of different grades of charcoal (pellets and granules) adhered onto the cryopanel. Detailed description of the experiment is further discussed.

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II. EXPERIMENTAL SETUP

Fig. 1 exhibits the experimental setup in the laboratory of Center for Cryogenic Technology (CCT), IISc Bengaluru. Rotary pump is used for the initial evacuation and continued further by turbo-molecular pump. The cryopanel is mounted over the Gafford McMahon (GM) cryocooler which uses helium gas as the working fluid [3]. The cryopanel is closed using radiation shields, and enclosed by Dewar chamber. This chamber will prevent the all the modes of heat losses to the surrounding. The cryocooler over which the panels are mounted makes the II stage where the temperature reaches around 4.2K as shown in Fig. 1.

The cryopanel is designed using the dimensions of commercially available Standard Varian cryopanel. The software used was solid works Fig. 2 shows the cryopanel designed for our experiment [4].

Seven panels made of copper material and four bolts were used to mount one cryopanel over another. Rotary pump is used for initial evacuation from initial pressure up to around 10^{-2} mbar. Then evacuation was done by switching to turbo-molecular pump which gives higher evacuation rate. There were separate sensors which will be mounted over the cold junction (near the cryopanel) and hot junction (outside the radiation shield) to monitor the temperature changes as the experiment was carried on.

Experiments were conducted for the following panels

- Bare cryopanel
- Bare cryopanel with adhesive
- Activated Charcoal (Pellets type) adhered over cryopanel
- Activated Charcoal (Granular type) adhered over cryopanel

III. EXPERIMENTAL PROCEDURE

The procedure recommended by the American Vacuum society (AVS 4.1) for the experiments were followed. The experimental procedure for all the panels remains the same which was described earlier. Rotary vane pump was used to create initial vacuum (10^{-2} mbar) of the system. Then the Turbo-Molecular pump was switched on for further vacuum in the range of 10^{-6} mbar. Once the required pressure was reached then the compressor is turned on. The static pressure and supply pressures are noted which indicates the compressor's performance indicators. Compressor sends the high-pressure cold air to the GM cryocooler which in-turn expands the air volume and helps in further temperature drop. The low-pressure air was sent back to the compressor which will compress the air again and the heat liberated while

compressing was taken out by the chiller unit. The three temperatures readings (Three sensors one over the cryopanel, one the below the cryopanel and third one on the cold head (first stage) are placed) for every ten minutes was noted down

which are the indicators of temperature drop in the first and second stages. The procedure was carried out until the temperature drops from an initial temperature of 273K to nearly 11K [5].

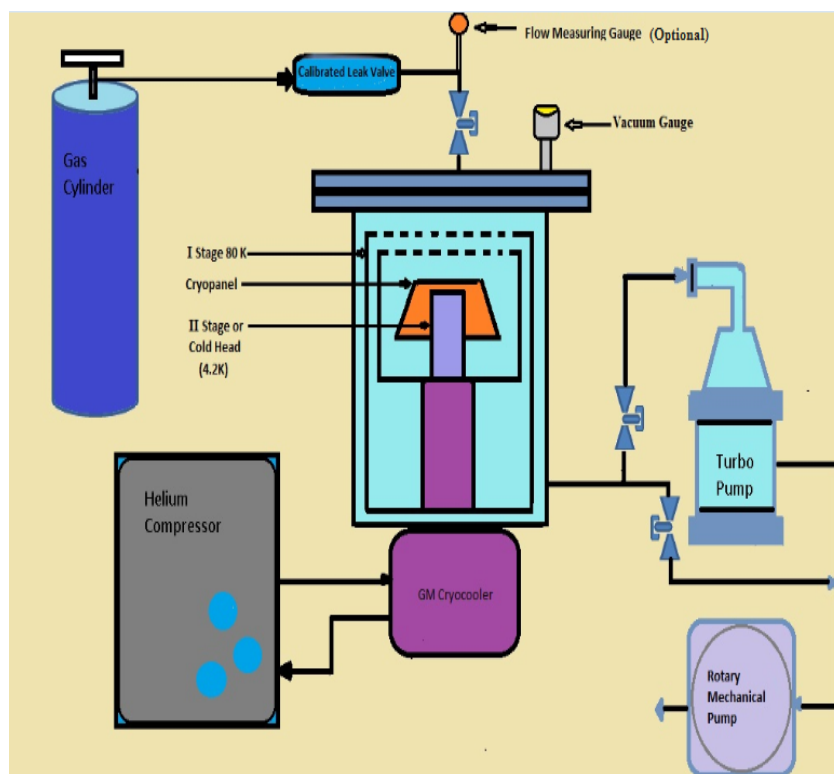


Fig. 1 Block diagram of Experimental setup

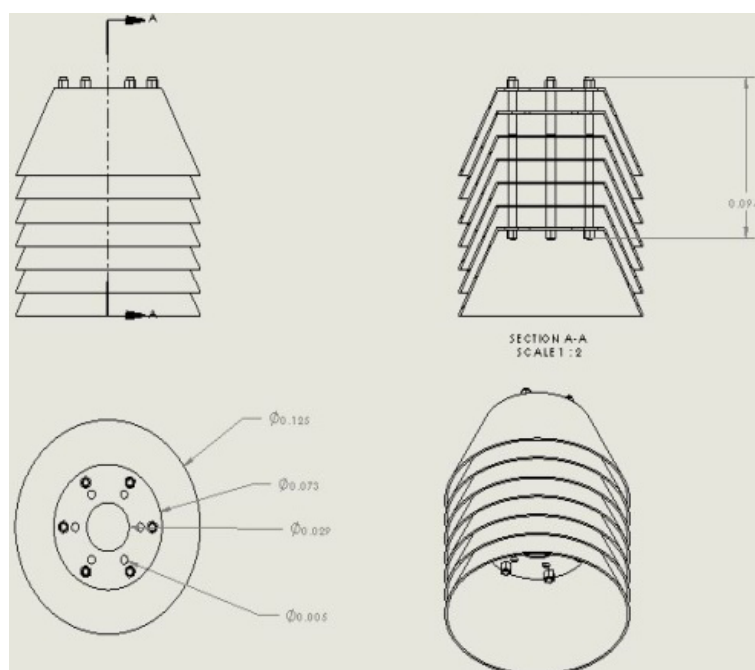


Fig. 2 Different sections of cryopanel



Fig. 3 Experimental Setup

The sample gas (He , H_2 , Ar , N) from the bellow was sent into the cryopanel through variable Leak Valve. The change in pressure and temperatures were noted down. The process was carried out until the temperature reading tends to increase. Next, the pumps were turned on in order to degas the cryo-chamber. Regeneration should be done in order to completely desorb the gas molecules adsorbed over the charcoal after the completion of each cycle [6].

IV. PUMPING SPEEDS MEASURED FOR BARE CRYOPANEL AND BARE PANEL WITH ADHESIVE



Fig. 4 Bare cryopanel mounted on the cryocooler

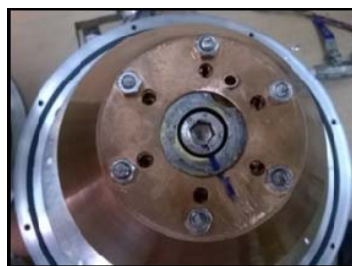
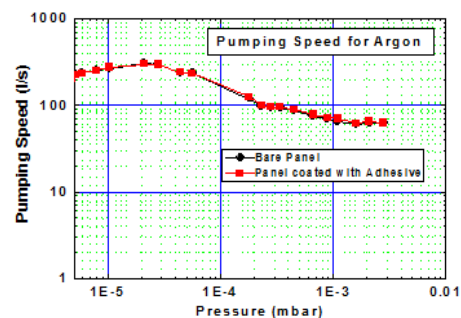
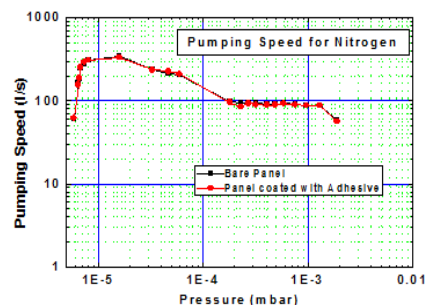


Fig. 5 Bare panel with adhesive mounted on cryocooler

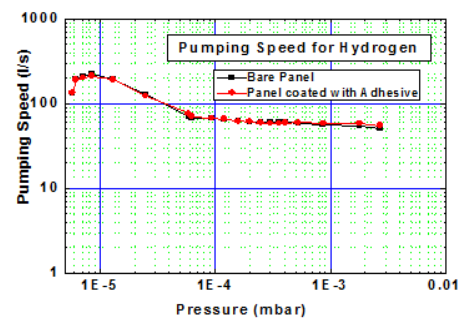
Bare cryopanel and epoxy resin with proper mixing ratio of hardener and epoxy over the bare panels (without using Activated carbon material) were tested by mounting them on the cryocooler individually in order to test the pumping performances [7]. The following plots were obtained for pumping speed for different gas species using bare panel and bare panel with adhesive in the cryosorption pump.



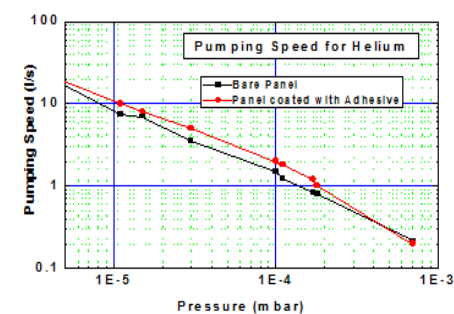
(a)



(b)



(c)



(d)

Fig. 6 (a) Pumping speed for Argon gas, (b) Pumping speed for Nitrogen gas, (c) Pumping speed for Hydrogen gas and (d) Pumping speed for Helium gas

A. Discussion on Results

In the case of bare panels, the measured pumping speed values were quite low compared to those of the commercial

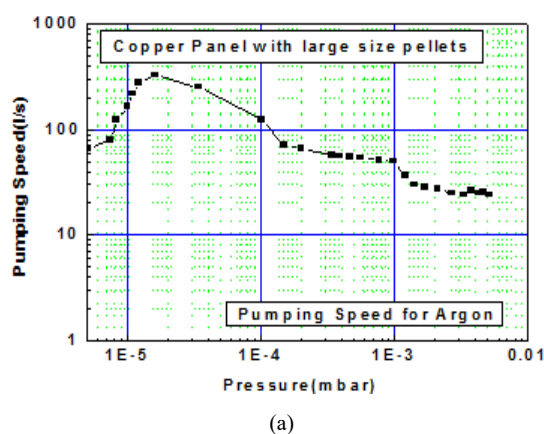
panel. Additionally, the bolts interconnecting the panels were made of stainless steel and hence, the bottom panels were warmer compared to the upper panels. Due to this, the performances especially in the case of hydrogen and helium were quite poor. In the case of helium, due to faster warm up of the lower panels, the pumping speed decreased rapidly with increasing pressure [8].

In the other case, the panels were coated with a layer of epoxy-based adhesive. Similar to the case of bare panels, the measured pumping speed values were quite low. The plots show the pumping speed values of the bare panels as well as those coated with a layer of adhesive. Likewise to the earlier case, the bolts interconnecting the panels were of stainless steel and hence, the bottom panels were warmer compared to the upper panels. Due to this, the performances for hydrogen and helium were quite low. For helium, the panels warm up faster, degrading the pumping performances.

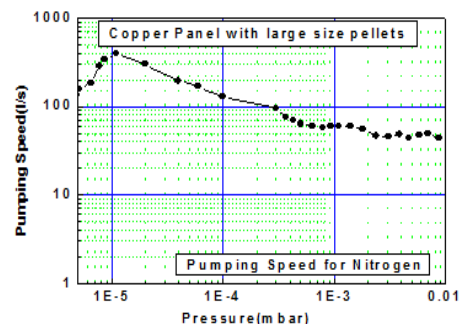
V. PUMPING SPEEDS MEASURED FOR ACTIVATED CARBON (PELLETS) ADHERED TO CRYOPANEL



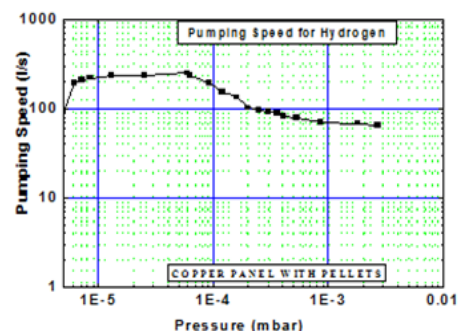
Fig. 7 Cryopanel with Activated Carbon



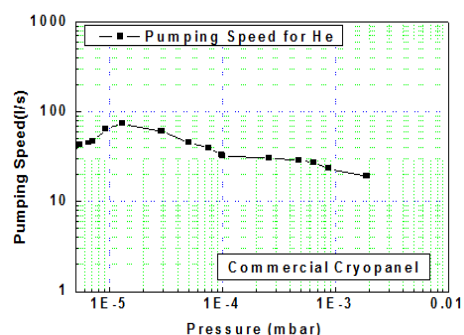
(a)



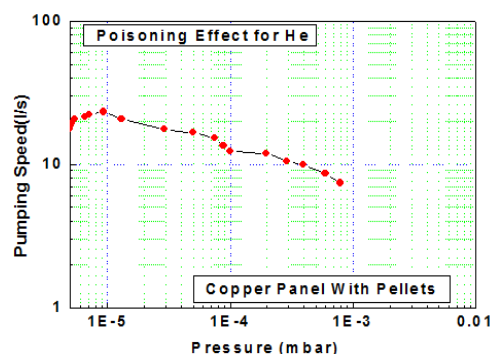
(b)



(c)



(d)



(e)

Fig. 8 (a) Pumping speed for Argon gas, (b) Pumping speed for Nitrogen gas, (c) Pumping speed for Hydrogen gas, (d) Pumping speed for Helium gas and (e) Poisoning effect for Helium gas

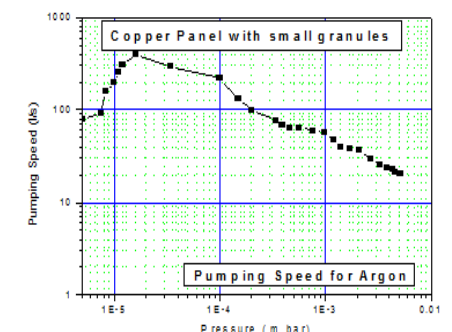
A. Discussion on the Results

Since the sizes of the pellets were large, they occupied more space and by this all the seven panels could not be accommodated in the cryocooler cold head. Thus, only four of the seven panels could be used in the available space. This has led to the reduction in the overall surface area of the AC panels. In addition, the temperature gradient across the panels was increased because of the large gap between the adjacent panels. However, the experiments were conducted with the stainless steel interconnecting bolts. In addition, this had the effect of increasing the gradient across the panels [9]. The panel temperature varied from ~ 10 K at the cold head to ~ 35 K at the farthest panel. Thus, the pumping speeds measured for Argon and nitrogen were also significantly lower as can be seen in the plots shown through Figs. 8 (a)-(e).

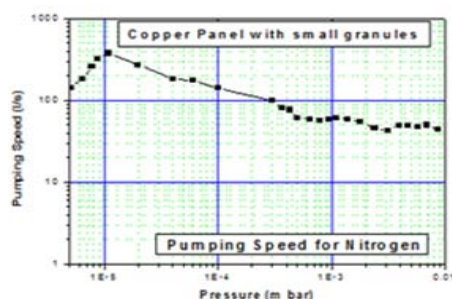
VI. PUMPING SPEEDS OF CRYOPANEL COATED WITH SMALLER SIZED AC GRANULES



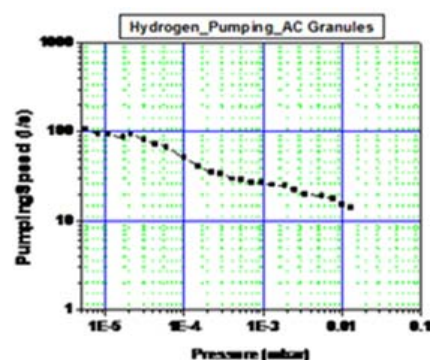
Fig. 9 Cryopanelcoated with smaller Activated Carbon



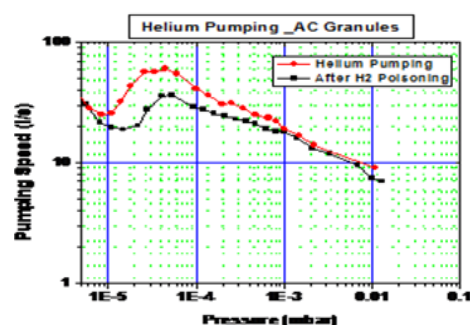
(a)



(b)



(c)



(d)

Fig. 10 (a) Pumping speed for Argon gases, (b) Pumping speed for Nitrogen gas, (c) Pumping speed for Hydrogen gas and (d) Pumping speed for Helium gas

A. Discussion on the Results

The sizes of the granules were small, they did not occupy much space, and thus all 7 panels could be accommodated in the cryocooler cold head. This led to the comparatively increase in the overall surface area of the panels. In addition, the temperature gradient across the panels did not increase because of much smaller gap between the adjacent panels. However, the experiments were conducted by changing the stainless steel interconnecting bolts with the new copper bolts. Due to these changes the pumping speed of Argon and Nitrogen were increased to an extent. But the results of Hydrogen and Helium were not satisfactory and Poisoning effect on Helium by Hydrogen gas showed a decrease in the pumping speed compared to Pumping speed of Helium gas alone [10].

TABLE I
COMPARISON OF RESULTS FOR ALL GASES AT DIFFERENT PARAMETERS

| Gases | A | B | C | D | E |
|---------------------------------------|-----|-----|-----|-----|-----|
| Argon | 622 | 308 | 302 | 330 | 410 |
| Nitrogen | 582 | 345 | 330 | 395 | 400 |
| Hydrogen | 328 | 220 | 210 | 248 | 289 |
| Helium | 512 | 20 | 24 | 70 | 80 |
| Poisoning Effect (Hydrogen on Helium) | 432 | - | - | 23 | 25 |

A: Varian Cryopump standard panel, B: Bare panel, C: Bare panel with adhesive, D: Panel with pellets, E: Panel with granules.

IV. CONCLUSIONS

The experiments conducted so far have given interesting results which would help in the further development of Cryosorption pumps in future. The Activated charcoal being the best-suited material was tested for various pumping speeds of different gas species. Bare panel and bare panel with adhesive are tested to understand the performances of the pump without adsorption. The results obtained using Varian cryopanel can be kept as a standard for comparing the experimental results obtained and the conclusions are drawn accordingly.

1. Bare panel and bare panel coated with epoxy based adhesive gave the similar performances.
2. Activated Charcoal with larger sized pellets gave good performance but indicated a drawback of low adhesion to the panel due to its size and also only four panels could be mounted over the cryocooler. Thus, more tolerance has to be given while designing the cryopanel such that all the seven panels could be mounted over the GM cryocooler.
3. Activated Charcoal panel with small sized granules gave very good performance since all the seven panels could be mounted over the cryocooler. Due to smaller pore sizes and numerous pores present in small granules there was considerable increase in adsorption. The AC density over the panels has to be increased further in order to get better performances.
4. Poisoning effect of Hydrogen over Helium can be seen in Standard Varian cryopanel and as well as on the small sized panels which shows a decrease in pumping speed of Helium gas.
5. For improved performance, AC coverage density should be improved considerably. Smaller sized granular charcoal performed better than bigger sized pellets.

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