Development of a cryogenic water-trap based on the recuperative mixture refrigeration cycle*

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In this paper, the details of a cryogenic water-trap based on the recuperative mixed-refrigerant refrigeration are disclosed. The cryogenic water-vapor trap is driven by an oil-lubricated single-stage compressor with nominal input power of 500 W. The lowest temperature is 110 K, at which the saturated vapor pressure of water is about 1e-10 Pa. A comparison was made between a vacuum system with and without the cryogenic water-trap. The result is that the pumping down time with the cryogenic water-trap can be reduced greatly as ten times. This will be important for most vacuum systems, e.g. vacuum deposition system, semiconductor industry, etc. Compared with the traditional cryogenic pump, there is no requirement of periodic maintenance. Moreover, this new developed cryogenic water-trap has high reliability, low cost, and easy to be built in large scale based on numerous merits of mixed-refrigerant refrigeration system.

INTRODUCTION

Water vapor is a major problem in most vacuum systems. It increases the pump-down time to reach the required base pressure. When the vacuum achieves 1e-4 Pa, more than 60 to 90 percent in residual gases is water vapor [1]. Traditional mechanical vacuum pumps including the molecular turbo-pump are not efficient in pumping water vapor. However, a cold interface at a low temperature range has a high water vapor pumping efficiency. This is the so-called cryogenic water-vapor trap. Indeed, the cryogenic water-vapor trap can not work independently. The work mechanism of the cryogenic water vapor pump is just using a cold interface to condensate the water from vapor phase to solid phase. So it is a passive working method. Usually, the water trap is used accompanied with an active vacuum pump such as the molecular turbo pump, oil diffusion pump, etc.

The temperature of the cold-trap interface varies from 110 K to 150 K based on the required vacuum associated with different base pressure required. The base pressure of the vacuum system is determined by the partial pressure of water vapor in the vacuum. Figure 1 shows relationship of between temperature and the partial pressure [2]. The equilibrium pressure of water is determined by the temperature. Therefore, the partial pressure of the water vapor varies from 1.0e-10 to 1.0e-7 Pa associated to the temperature range from 110 to 150 K. This required temperature range is just suitable for the low temperature mixed-refrigerant refrigerator. Therefore, a low temperature water-vapor trap was developed based on a mixed-refrigerant Joule-Thomson refrigerator. The details of the design and test performance of a cryogenic water-vapor trap using a mixed-refrigerant refrigerator will be presented in this paper.

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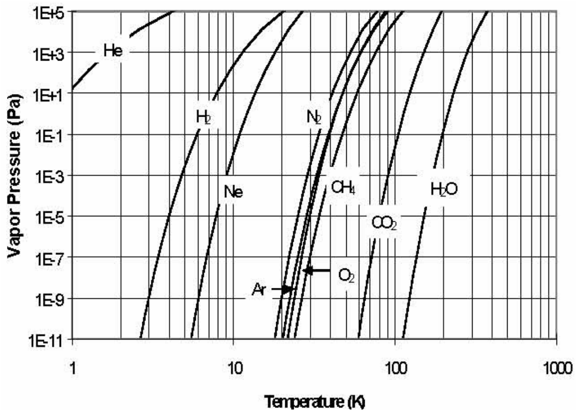
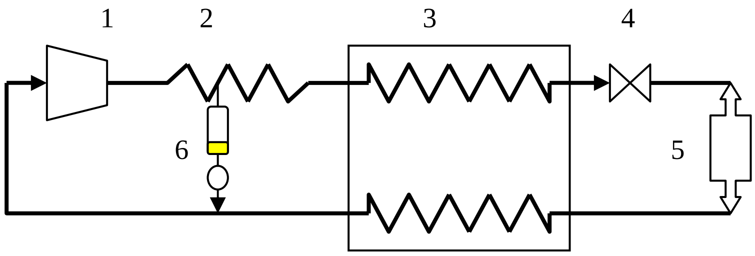


Figure 1 Equilibrium vapor pressure curves for common gases



1: Compressor, 2: After cooler, 3: Recuperator, 4: Throttle valve, 5: Evaporator- cold trap interface, 6: oil separator

Figure 2 Schematic diagram of the refrigerator

DESIGN OF THE CRYOGENIC WATER TRAP

Design of the mixed-refrigeration subsystem

The refrigeration power required at the temperature range described above is different for different vacuum vessel size, which determines the vacuum pump used. In this experiment, a standard vacuum test setup with a nominal inner diameter of 160 mm is used to study the performance of the new developed cryogenic water vapor trap. Therefore, a suitable mixed-gases Joule-Thomson refrigerator was used to improve the evacuation performance by removing the extra water vapor in the vacuum system. Figure 2 shows the schematic diagram of the refrigerator. The compressor used in this refrigerator has a nominal input power of 500 W. A tubes-in-tube heat exchanger was used as the recuperator. A capillary tube is used as the throttle valve. The evaporator is designed as the cold trap interface, which was fitted in the inlet of the turbo vacuum pump. An optimal mixture with six components was used as the refrigerant. The details of the refrigerator can be found in the reference [3].

Configuration of the cryogenic water trap system

A new configuration was developed in this cryogenic water trap system. Figure 3 shows the schematic diagram of the system. In this system configuration, the cold trap interface which is also acting as the evaporator in the refrigeration system, was put in the vacuum passage between the inlet of the vacuum pump and the outlet of vacuum vessel. The cold stage of the refrigeration system mainly consisting of the recuperator is put in a vacuum Dewar, which is evacuated by the first stage pump. On the other hand, the cold stage of the recuperator, in which the coldest temperature is almost as low as the cold trap interface, provides another cold trap for the first stage vacuum pump*. This obviously decreases the pressure after the turbo pump, and improves the performance of the vacuum system greatly.

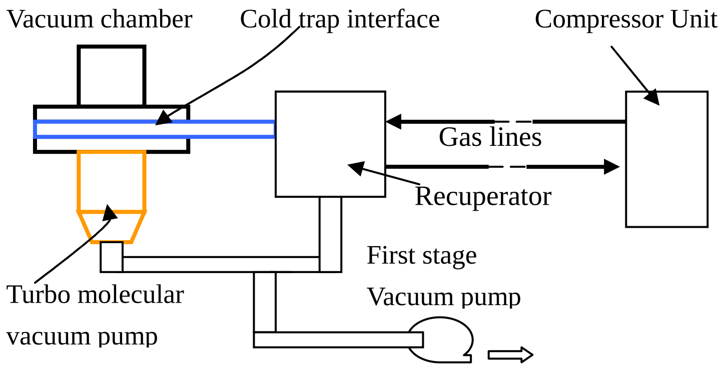


Figure 3 Schematic diagram of the system

Figure 4 Photo of the system

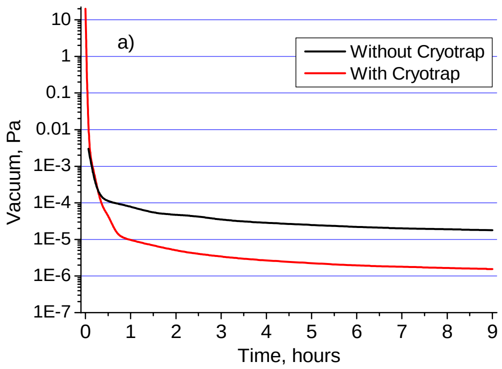
* Patent pending

Figure 5 Photo of the cold trap interface

Figure 6 Configuration of the trap interface

EXPERIMENTAL TEST RESULTS

A prototype of the cryogenic water vapor trap, based on the design consideration described above, is developed and tested. Figures 4, 5 and 6 show the photos of the prototype and the cold trap interface. This prototype was tested comprehensively to know its performance. Comparisons of the pumping speed and the ultimate pressure of the system were made with and without the cryogenic water vapor trap. Figure 7 shows the results of the comparison. From Figure 7 a), it is obvious that the pump speed with the cryogenic trap is almost about ten times faster than that without the cryogenic trap. Moreover, the ultimate vacuum pressure is also ten times lower than that without the cryogenic trap. The improvement of the performance is same for a similar cryogenic water-vapor trap – Aquatrap from Polycold Company [4, 5], which is shown in Figure 7 b). The temperature cool-down curve of the cold interface is shown in Figure 8 as well as the variation of the first stage pressures at two situations that with or without the cold trap.



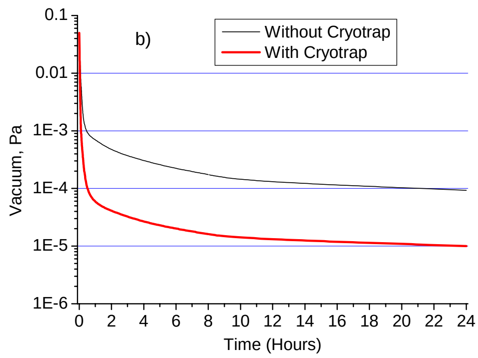
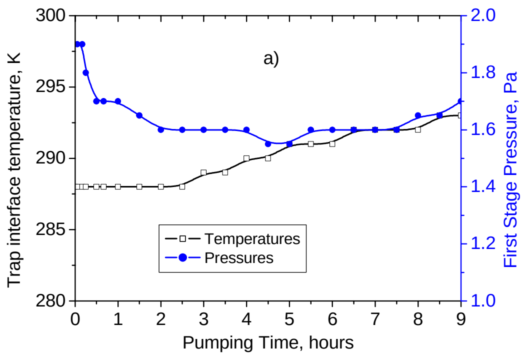


Figure 7 Vacuum performance of the cryo-trap, a) prototype built in this work, b) Aquatrap [5]



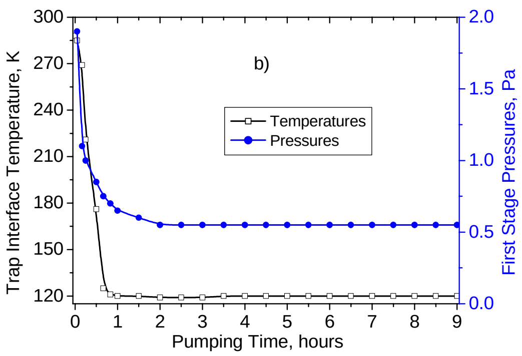


Figure 8 Variations of interface temperature and the first stage pressures, a) without b) with cold trap

In the prototype built in this work, when the cryogenic trap does not operate, the first stage steady operation pressure is about 1.6 Pa, and the vacuum ultimate pressure is 2.0e-5 Pa after 9 hours operation. However, when the cryogenic trap works, the first stage pressure decreases down to about 0.55 Pa in the final steady state operation, and the ultimate pressure reaches up to 1.6e-6 Pa. The decease of the first stage pressure can improve the performance of the turbo-molecular pump. The final temperature of the trap interface is about 120 K in the steady state operation.

The vacuum system pumped by a turbo-molecular pump combined with a low cost mixed-refrigerant Joule-Thomson refrigerator can provide the similar evacuating performance as a cryogenic pump. The traditional cryogenic pump evacuates the vessel by capturing gases with a colder interface which is cooled by a cryogenic refrigerator e.g. GM cryocooler at a cryogenic temperature as low as 20 K. Therefore, the built cost of a cryogenic pump is much larger than the vacuum system consists of a turbo pump and a cryogenic water trap. Moreover, the cryogenic pump needs regular maintenance, e.g. replacing the oil removing unit in the cryocooler compressor. However, the built cost cryogenic water-vapor trap cooled by a mixed-refrigerant Joule-Thomson Refrigerator is very low because of the usage of large-scale commercial compressor widely used in room temperature refrigeration field. Seldom maintenance is needed in a long operation period.

SUMMARY AND DISCUSSION

A cryogenic water-vapor trap based on the mixed-gases Joule-Thomson refrigerator was designed, built, and tested in this paper. The overall pumping speed of the system that combined with a turbo molecular pump is higher ten times than that without the cold trap. The cryogenic water-vapor trap presented in this work offers the highest pump speed for water vapor, and the lowest cost. When used with other high vacuum pumps, it offers the best overall performance available today for large vacuum systems operating in the range from atmosphere to 1.0e-6 Pa.

Indeed, all vacuum pumps including mechanical, oil diffusion, cryopump, ion, titanium sublimation, etc., have limitations. These limitations include cost, contamination, operational difficulty, selective gas pumping, pumping speed, pressure range and throughput. When attempting to select the high vacuum pump for any given application, it becomes obvious that there is no universal or ideal pump. The practice is to make serious compromises usually based on cost trade-offs. The proper approach is to utilize the advantages of the most applicable pumps and use a combination of pumps. The cryogenic water-vapor presented in this work combined with a turbo pump has many advantages over other kind pump include cost, availability, ease of operation, and faster response time in cool-down. They are easy to install on existing vacuum systems; it only requires locating a cryogenic interface on the chamber.

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