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Tempelmeyer

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[54] **CRYOSORPTION PUMPING WITH FROST SORBENTS**

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[51] **Int. Cl.** **B01d 5/00**

[58] **Field of Search** 62/55.5

[56] **References Cited**

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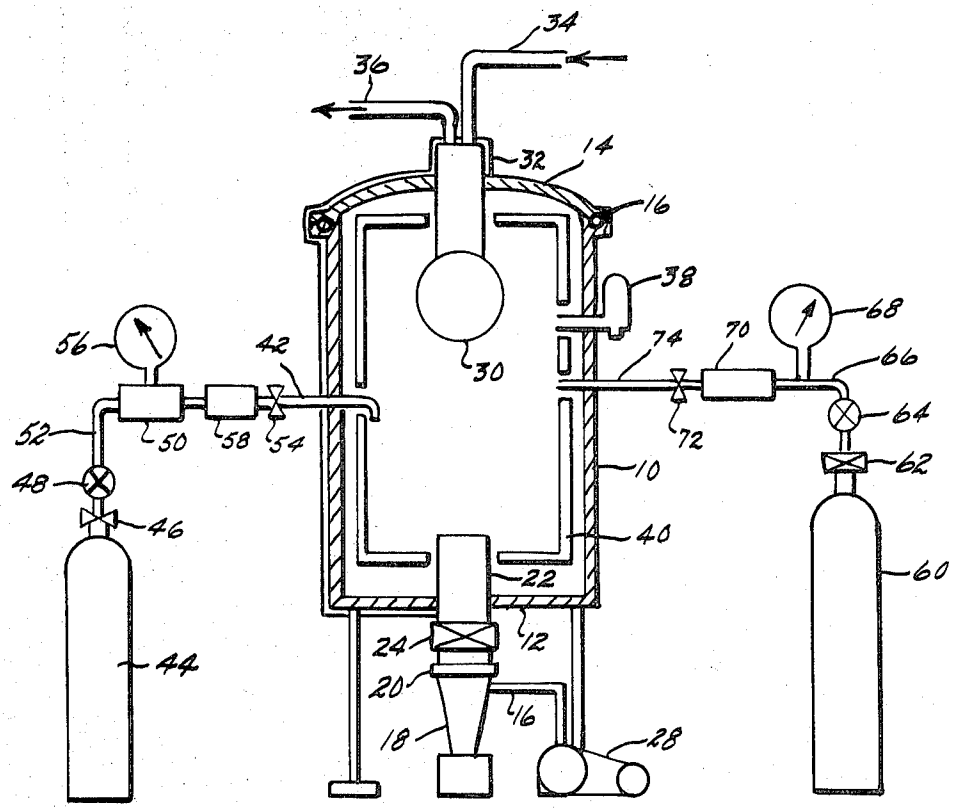
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Primary Examiner—William J. Wye

[57] **ABSTRACT**

A system for providing very high vacuums in closed vessels having a hydrogen, helium or neon gaseous atmosphere. The gaseous atmosphere is placed in communication with a cryogenically cooled frost coated structure. The frost is formed on the cooled surface by depositing a sorbent gas, such as carbon dioxide, sulfurdioxide or methyl chloride onto the surface under processing conditions that produce a disordered or amorphous frost structure. The formation technique utilized in forming the frost provides a structure that adsorbs hydrogen, helium or neon gas molecules as they come in contact with the frost surface to a degree not heretofore achieved. As a result, the hydrogen, helium or neon atmosphere is, in effect, pumped out of the closed vessel resulting in the production of a very high vacuum therein.

3 Claims, 2 Drawing Figures



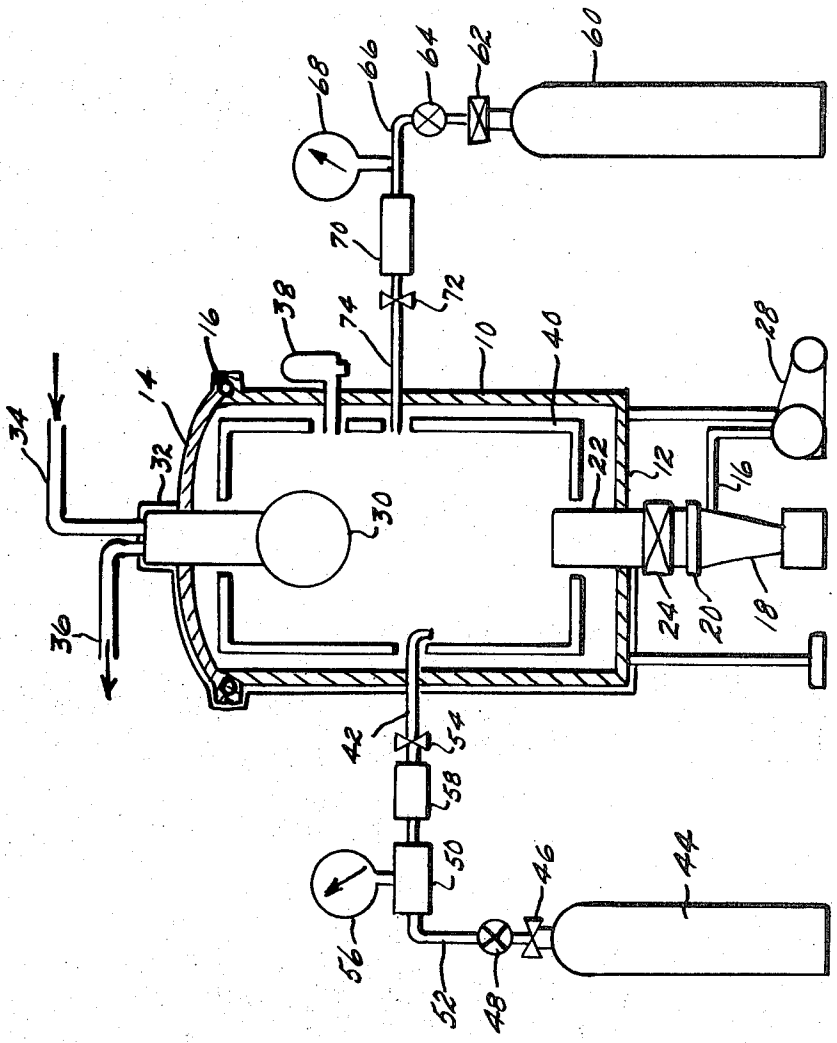

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FIG. 1

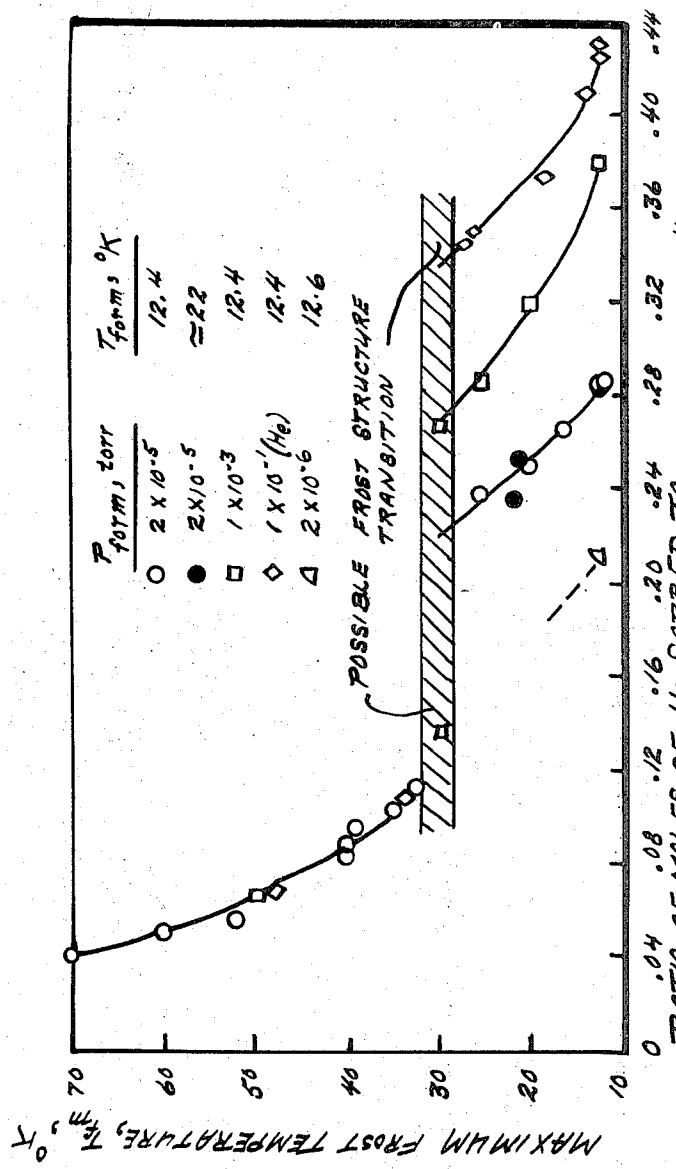

MAXIMUM FROST TEMPERATURE, T_m, °K
70
60
50
40
30
20
10
0
.04
.08
.12
.16
.20
.24
.28
.32
.36
.40
.44
RATIO OF MOLES OF H_2 SORBED TO MOLES OF CO_2 IN FROST AT $P=3\times10^{-4}$ torr
P_{form}, torr
T_{form}, °K
○ 2×10^{-5} 12.4
● 2×10^{-5} ≈22
□ 1×10^{-3} 12.4
◇ 1×10^{-1} (H_2) 12.4
△ 2×10^{-6} 12.6
POSSIBLE FROST STRUCTURE TRANSITION

FIG. 2

CRYOSORPTION PUMPING WITH FROST SORBENTS

BACKGROUND OF THE INVENTION

This invention relates to an improved system for producing a high vacuum by cryosorption pumping. In a more particular aspect, this invention relates to a system for producing high vacuums by effecting the sorption pumping of gaseous materials through the utilization of a cryodeposited frost.

Various fields of technolocy utilize low pressure systems as an integral part of their operational capability. The production of very low pressure or high vacuums, however, poses a problem since the vacuum systems heretofore suggested have not proven effective in attaining pressures on the order of 1×10^{-10} millimeters of mercury or for removing quantities of hydrogen, helium or neon from vacuum chambers at higher pressures. Diffusion pumps, either alone or in series, and getter pumps have been suggested as a means for achieving very low pressures. These systems, however, have not proved effective with the result that a system referred to as cryopumping has come into widespread use. In this system, high vacuums are produced by condensing a gas onto a cryocooled surface whose temperature is below the condensation temperature of the gas. Extremely low ultimate vacuums are produced by this system and it requires less power than the conventional high vacuum diffusion pump.

The reduced power consumption of a cryopump system, as well as its ability to produce very high vacuums, makes cryopumping especially desirable for a variety of industrial applications. However, space technology applications present an additional problem since the vacuum chambers and space simulation chambers used in this particular discipline contain gases that are not condensible at the low temperatures and pressures encountered in cryopumping. Cryopumping is not economically feasible for the removal of hydrogen, helium and neon because of the very low condensation temperature exhibited by these gases at low pressures. Neon gas does not generally present a problem because of its rarity, however, hydrogen and helium are present in most vacuum systems and often limit the ultimate pressure which may be achieved.

The problem of removing large amounts of hydrogen is of particular concern because of the increasing need to evaluate the operation of small space propulsion units in ground-based space chambers. A number of tests have been conducted but the maximum altitude which may be simulated during such tests is goveren by the amount of hydrogen introduced into the chamber from the rocket engine and has been generally limited to a simulated 200,000 to 250,000 ft.

Hydrogen has been pumped heretofore by methods that include the use of conventional diffusion pumps, turbo-pumps, ion pumps and others. Each offers certain advantages, but each also has a limited pumping speed or capacity for this gas. The use of diffusion pumps is limited by the amount of chamber wall space available for mounting the pumps. The use of molecular turbo-pumps as well as various types of ion pumps is limited by the low pumping speeds exhibited by these pumps. Various types of sorption pumps also have been employed in which either physical or chemical adsorption are utilized. For example, titanium films are widely used to chemically absorb hydrogen. However, the titanium film often times becomes saturated by other gases (mainly nitrogen) and, as a result, is ineffective for pumping hydrogen.

In attempting to overcome the problems previously encountered in the pumping of hydrogen, helium and other relatively noncondensible gases, it was found that a system that included the formation of a cryodeposited frost formed from condensing certain gases onto a cryogenically cooled surface provided an effective system for pumping these non-condensible gases. The system is referred to as cryosorption pumping and relates to a process wherein a sorbent (the gas being condensed on a cryogenically cooled surface) is cooled by a cryogenic fluid to a temperature above the normal condensation temperature of the sorbate (the gas being pumped out of a closed vessel to achieve very low pressures).

Cryosorption pumping is a well known phenomenon and many have studied the formation of cryodeposited frosts. However, only a few studies of the cryosorption properties of cryodeposits have been carried out. For example, the pumping of various cryofrosts for both hydrogen and helium have been reported, but the exact pumping mechanism and the factors which influence it are essentially unknown. With the present invention, however, it has been found that certain factors are critical if especially efficient cryosorption pumping system is to be achieved. Adherence to these factors results in the unexpected production of very low vacuums on the order of 1×10^{-10} mm of mercury. It is now known that the sorption capacity of cryodeposited frosts depends upon the conditions under which the frost was formed and its temperature history. The criticality of various processing parameters has been discovered and adherence to these parameters during the pumping operation leads to the formation of a disordered or amorphous frost structure. Such a structure, as opposed to an ordered or porous structure, unexpectedly exhibits a much greater equilibrium sorption capacity for hydrogen or helium than was heretofore achieved by known cryosorption pumping systems.

SUMMARY OF THE INVENTION

In accordance with this invention, it has been found that extremely high vacuums can be produced in closed vessels by a system which pumps out gases contained in the closed vessels. The system includes the step of condensing a gaseous material onto a cryogenically cooled surface in the form of a frost. After frost formation, the gaseous atmosphere contained within the the closed vessel is placed in communication with the cryodeposited frost. The frost adsorbs the gaseous molecules of hydrogen, helium or neon, thereby creating a low pressure atmosphere within the closed vessel. Apparently the surface diffusion of adsorbed gas molecules into the frost structure is the basic pumping mechanism. The sorption capacity of the frost depends upon the conditions under which the frost was formed and frosts formed in a manner to make them amorphous or disordered, as opposed to a porous or ordered structure, provide greater sorption capabilities than frosts formed in accordance with previously known techniques.

Although the particular processing steps for cryopumping gaseous materials, such as hydrogen, helium or neon are well known, the prior art was not aware of

the particular processing parameters needed to produce extremely high vacuums. The present invention, however, has discovered that adherence to certain factors during the frost formation and cryosorption pumping processes will produce vacuums of a degree much higher than that heretofore achieved. The frost sorption capacities of the cryodeposited frosts of this invention are increased by a factor of 10 or more as compared to the sorption capacities of frosts formed in accordance with previously known techniques.

The particular processing parameters found to be critical include the temperature of the surface upon which the frost is formed, the rate at which the frost is formed and the pressure level during frost formation. The frost thickness and the frost temperature during the pumping operation are also essential. The frost specie, that is the particular gaseous material used to form the frost, also constitutes a critical factor, as does the stike rate at which the frost forming gaseous material is deposited upon the cryosurface. Adherence to these processing parameters during the sorption pumping of hydrogen, helium or neon from a closed vessel permits the attainment of extremely high vacuums within the closed vessel. These vacuums are especially useful for creating a simulated space atmosphere for the testing of space vehicles and their component parts.

Accordingly, the primary object of this invention is to provide a method for producing extremely high vacuums.

Another object of this invention is to provide a method for evacuating a gaseous material from a closed vessel by placing the vessel's gaseous atmosphere in communication with a surface cryogenically cooled to temperatures below 30° K.

The above and still other objects and advantages of the present invention will become more readily apparent upon consideration of the following description thereof when taken in conjunction with the accompanying drawings:

In the drawings:

FIG. 1 represents a schematic view of a system suitable for use with this invention, and

FIG. 2 is a graohic representation showing sorption capacities of frosts formed in accordance with the invention.

DESCRIPTION OF THE PRESENT EMBODIMENT

The present invention encompasses a method for producing extremely high vacuums through the utilization of a cryodeposited frost. Although the prior art was aware of the processing steps needed to create vacuums through the use of such frosts, they did not know nor appreciate the criticality of certain processing parameters for producing very high vacuums. The present invention, however, has discovered the critical nature of certain parameters.

In general, the process includes the step of passing a gaseous material contained within a closed vessel over a cryodeposited frost which, in turn, adsorbs the molecules of the gas, thereby producing a pumping action that creates a vacuum within the closed vessel. In order to achieve the extremely high vacuums of the present invention, however, it is necessary that certain processing parameters be adhered to during the pumping operation. The temperature of the surface upon which the frost is formed is critical. It should be as low as possible with a maximum temperature of 25° K for pumping hydrogen or neon, and 5° K for pumping helium. The rate at which the frost is formed is also critical with the frost having a minimum growth rate of about **0.1** microns per minute. The pressure level during frost formation should be maintained at a minimum of 1×10^{-4} mm of mercury. The frost thickness is also critical with an optimum thickness being from 1 to 4 microns with a maximum thickness not to exceed above 1 mm. The frost temperature during pumping is also a critical factor with an optimum temperature being as low as possible, and a maximum temperature being 22° K for hydrogen pumping, and at 5° K for helium pumping. The frost specie, that is the particular type of material used to form the cryodeposited frost, is also critical. Carbon dioxide, sulpher dioxide, nitrous oxide, methylchloride, carbon tetrachloride, argon, and nitrogen, in that order of preference, have been found to produce the highest vacuums.

Inasmuch as the terminology employed to describe the cryosorption pumping phenomena is not used consistently in the literature and because the similarity of terms can often give rise to confusion, it is believed adviseable at the onset to provide a number of definitions. Adsorption describes a phenomenon in which the molecules striking a surface adhere to it and remain for a finite time. Surfaces may be covered with monolayers or multiple layers of molecules due to adsorption. Absorption is used to describe the migration of adsorbed molecules away from their adsorption sites and into the interior of the material. Adsorption may occur by interstitial diffusion through a crystal structure or by surface diffusion into pores, cracks, grain boundaries, and so forth which lead into the interior of the material. Molecules which are adsorbed onto a surface might then be absorbed into the material. Sorption is often used as a general term to describe adsorption and/or absorption processes where the adsorption may be either physical or chemical. This term is frequently used to represent a combined adsorption-absorption phenomenon as well as to describe some sort of adsorption/absorption phenomenon which is not well defined.

The sorbate is the gas being sorbed by a sorbent. For example, in the present invention, hydrogen is the sorbate while the cryodeposited frost is the sorbent.

The term condensation temperature is used herein to describe the temperature at which a large quantity of gas would be condensed and cryopumped at a pressure of about 10^{-7} torr. However, the condensation temperature changes only slightly with orders of magnitude changes in the pressure. Generally, it is about 5° K lower than is specified by the vapor-pressure curve for the gas in question, or about 80° K for CO_2, 26° K for argon, 23° K for N_2, and 5° K for H_2.

The term Cryopumping describes a process in which pumping is achieved by condensing a gas onto a cryogenically-cooled surface whose temperature is below the condensation temperature. It is, in effect, a special form of adsorption in which the surface acts as an energy sink and greatly decreases the probability of desorption. The term cryosorption pumping, however, is used to describe a sorption process in which the sorbent is cooled by a cryogenic fluid to a temperature above the normal condensation temperature of the gas being pumped. Cryotrapping describes a phenomenon in which a non-condensible gas is swept onto a cryosurface by a condensing gas and buried and trapped there

by the condensed gas. The present invention is concerned with the cryosorption pumping phenomena.

Now, with reference to FIG. 1 of the drawing, there is disclosed a system for carrying out the method of this invention. In general, it comprises a conventional vacuum chamber **10** having a stainless steel wall, a flat bottom **12,** and a top **14** which is removable. The top **14** is mated to the wall at flange **16** by either a copper shear-seal gasket or an O-ring, not shown. The chamber **10** is normally pumped out by a conventional diffusion pump **18** equipped with a liquid nitrogen cooled cold trap **20** and communicates to the chamber **10** through duct **22.** The chamber **10** can be isolated from the pumping system by valve **24.** The diffusion pump **18** is connected through line **16** to a backing mechanical pump **28.** A cryopumping surface **30** with a vacuum jacket **32,** supply line **34** and return line **36** are installed in the chamber **10** and attached to the removable lid **14** of the chamber **10.** Although the cryosurface **30** is spherically shaped, its particular configuration and location is not critical. It is cooled to a predetermined temperature by using cold gaseous helium from a refrigerator or dewar, neither of which is shown. The chamber pressure is measured by a suitable gage **38.** The chamber also contains a cylindrical internal liner **40** which serves as a radiation shield to reduce the heat load on the cryosurface **30.** The latter also serves as the surface upon which the frost cryosorbent is deposited. The mechanical pump **28** is used to initially reduce the chamber pressure to a few microns of mercury. Then, either the diffusion pump **18,** or the cryosurface **30** which has been cooled with gaseous helium, is activated and the chamber pumped to 1×10^{-6} mm mercury. Then the chamber is isolated from the external pumping system by valve **24,** and then either carbon dioxide, sulfur dioxide, or some other sorbent gas is caused to flow into the chamber **10** through line **42.** The sorbent gas is obtained from commercial cylinders **44** and passes through a valve **46** and pressure regulator **48.** It then passes to a surge tank **50** by means of line **52,** and then through an isolation valve **54.** The pressure in surge tank **50** is set by the regulator **48** and read by gage **56** such that a given flow rate of the sorbent gas passes through the sintered steel leak **58.** When the frost is formed, the chamber pressure is decreased to as low as 1×10^{-9} Torr. Also, large amounts of hydrogen can be added to the chamber and the pressure level maintained as the frost adsorbs the hydrogen.

In some instances, the frost sorbent is formed by evacuating the chamber **10** by means of the diffusion pump **18,** isolating it by valve **24** and then backfilling with helium gas supplied by a bottle **60.** The helium is introduced into the chamber through a valve **62** and pressure regulater **64.** The pressure in a section of the supply line **66** is read with a pressure gage **68.** The helium then passes through a surge tank **70,** a shut-off valve **72,** and enters the chamber **10** by means of line **74.** After the chamber **10** is repressurized with helium to a pressure of from 1×10^{-3} mm mercury to **1** mm mercury, the cryosurface **30** is cooled and the sorbent gas is admitted to the chamber **10** by way of line **42** and a specified thickness of frost is formed on surface **30.** The helium is then removed from the chamber by means of the diffusion pump **18** and the chamber pressure decreased by 1×10^{-9} mm mercury. The frost cryosorbent formed in this manner will adsorb very large quantities of hydrogen when it is subsequently introduced into the chamber.

The apparatus illustrated in FIG. **1** operates in the following manner.

The chamber **10** is pumped to the lowest pressure possible by means of the mechanical pump **28.** Then, either the diffusion pump **18** or the cryopump **30** is used to reduce the pressure reading on gage **38** to about 1×10^{-6} mm mercury. If hydrogen is the expected major residual gas, then the cryosurface **30** is used and cooled with gaseous helium to a temperature from 12° to 16° K. If helium is expected to be the residual gas, the diffusion pump **18** is first used to reduce the pressure to the 10^{-6} mm mercury range and then valved off from the chamber **10** after which the cryosurface **30** is cooled with liquid helium to 4.2° K from a dewar, not shown. In either case, as the cryosurface **30** is cooled, the carbon dioxide or some other specie sorbent gas is admitted to the chamber **10** by line **42** and a frost of **1** to **2** micron thickness is formed on the cryosurface **30.** The sorbent gas is admitted at as high a rate as possible consistent with keeping the cryosurface temperature in the desired range. The sorbent addition rate depends upon the available refrigeration capacity; however, it is very important to employ as high a formation rate as possible but not allow the cryosurface temperature to increase above 20° K for hydrogen pumping or 5° K for helium pumping. Frosts formed in this manner are disordered or amorphous and possess superior pumping characteristics.

Carbon dioxide addition rates of as high as 3×10^{17} molecules/sec- cm^2 (striking the cryosurface) which correspond to a frost growth rate of about 1 μ/min have been used, and resulted in pumping of one hydrogen molecule for each three carbon dioxide molecules added to the system. Higher frost formation rates would be beneficial and could be employed in systems with larger refrigeration capacities. The chamber pressures which can be achieved by the frost cryosorption pumps were about 1×10^{-9} mm mercury when the frost is formed on a gaseous helium cooled surface and about 1×10^{-10} mm mercury when it is formed on a liquid helium cooled surface.

In the pumping of continuous hydrogen gas additions, the system is pumped down in the same way as pointed out above. After the pressure reaches about 1×10^{-6} mm mercury, the diffusion pump **18** is isolated from the system by closing valve **24.** Then the chamber is backfilled with helium and the cryosurface **30** is cooled to as low a temperature as possible. The pressure level to which the chamber **10** is backfilled depends upon the available refrigeration capacity. It should be as high as possible consistent with keeping a gaseous helium cooled cryosurface below 20°K. Pressure levels from 10^{-2} mm mercury to 1 mm mercury are readily possible. After the helium flow is terminated, the carbon dioxide or other specie sorbent gas, is admitted into the chamber **10** through line **42** at as rapid a rate as possible consistent with maintaining the above noted cryosurface temperature. The amount of carbon dioxide gas added depends upon the amount of hydrogen to be pumped. About **2** moles of carbon dioxide will pump one mole of hydrogen if the frost is cooled to below about 14° K. This capacity is known to hold for frost thicknesses up to **10** microns. After the frost is formed, the helium is removed by means of diffusion pump **18.** The frost cryosorbent is now ready to

pump any hydrogen additions to the chamber **10** at a speed of 25 to 30 liters/second for each square centimeter of frost area, and maintain a pressure level between 10^{-6} to 10^{-8} mm Hg depending upon the hydrogen addition rate.

This pumping arrangement is useful for proton accelerators, space chamber rocket engine tests or any type of space simulation test installation in which sizable amounts of hydrogen must be pumped and low pressures maintained. Once the frost layer nears the saturation point, additional carbon dioxide or other sorbent gas, may be added as another layer of frost on top of the first and the pumping will continue.

The pumping of continuous helium gas additions is accomplished in the system by pumping the system with the mechanical pump **28** and the diffusion pump **18** to about 10^{-6} mm mercury. Then the cryosurface **30** is cooled with liquid helium and the chamber pressure is decreased to about 10^{-8} mm mercury as the residual hydrogen is cryopumped. Next, carbon dioxide, or other sorbent gas, is added in the manner described above and cryopumped on the cold surface **30.** The sorbent addition rate should be as high as possible consistent with maintaining a cryosurface temperature below 5° K. The amount of helium which will be pumped depends on the amount of carbon dioxide added. About **6** moles of carbon dioxide will pump one mole of helium if the frost is at 4.2° K. One may also supercool the liquid helium by evacuating the return line **36** with a mechanical pump (not shown in FIG. **1**) and produce lower frost temperatures and greater sorption capacities. A carbon dioxide frost cryosorbent will pump helium at a rate of about 10 liters/second for each square centimeter of frost area.

After the gaseous helium cooled carbon dioxide frost sorbent has been saturated with hydrogen, it may be desorbed and re-used if an auxiliary pumping system, such as pumps **18** and **28,** is available. Such a procedure is highly desirable when the hydrogen gas load to be pumped is or can be made intermittent. In this procedure, the pumping system isolation valve **24** is opened and the cryosurface allowed to warm at the rate of 1° k/minute until the temperature reaches approximately 22° K. At this point nearly all of the hydrogen will be desorbed from the frost and removed by the pumping system. This procedure minimizes structural changes in the frost which adversely affect its subsequent pumping performance. The frost temperature should never be allowed to increase above 30K. Valve **24** is again closed and the cryosurface **30** cooled back down to the original temperature whereupon it is ready to again pump hydrogen. This process may be repeated over and over.

Cryodeposited frosts formed by condensing certain gases, such as those enumerated heretofore, on surfaces cooled to temperatures between 10° and 20° K have been shown to provide an effective means of pumping hydrogen through the use of the cryosorption pumping phenomena. Moreover, these frosts will also effectively pump helium if the cryosurface temperature is maintained at 4.2° K and below. Frost cryosorption pumping offers a number of advantages over the use of previously known methods for pumping hydrogen, neon or helium. It possesses higher pumping speeds per unit area and higher sorption capacities per unit volume than do the metal film or molecular sieve systems. It is readily cooled and kept cold in contrast to a molecular sieve system. A fresh frost layer can be formed over one that has been previously saturated providing for a continuous operation. Providing a fresh frost layer does not introduce as large a heat load on the system as occurs when a titanium metal is deposited in the metal film system. Also, it is not as subject to contamination by other gases as are titanium films.

FIG. **2** provides a summary of maximum equilibrium sorption capacities of carbon dioxide frosts at a temperature of 12.4° C as a function of the intermediate warming temperature.

Some typical values of frost conditions at which the carbon dioxide frost is formed are summarized in Table I.

TABLE I

Chamber pressure during formation P form Torr	CO_2 Strike Rate	Frost Growth Rate
2×10^{-6}	6.3×10^{14} mol/cm²-sec	0.0045μ/min
2×10^{-5}	6.3×10^{15} mol/cm²-sec	0.045μ/min
2×10^{-4}	6.3×10^{16} mol/cm²-sec	0.45μ/min
1×10^{-3}	$3/15 \times 10^{17}$ mol/cm²-sec	1.0μ/min

To summarize briefly, the present system utilizes certain critical parameters during the pumping operation in order to achieve very high vacuums. These parameters are outlined as follows: First, the sorbent species found to be effective may be selected from a group of gaseous materials consisting of carbon dioxide, sulfur diode, nitrous oxide, methyl chloride, carbon tetrachloride, argon and nitrogen. Secondly, cryosurface temperatures are preferably between 5° K to 25° K for pumping hydrogen and 2° K to 5° K for pumping helium. Thirdly, the frost temperature must never exceed 30° K, and is preferably between 10° K to 22° K for hydrogen and 2° K to 5° K for helium. Other parameters include sorbent strike rate as high as possible and frost thicknesses not to exceed **1** mm but preferably being about from 0.02 to 4.0 microns. The frost should be formed as rapidly as possible consistent with maintaining frost temperatures in the ranges outlined above. If frost is formed with the chamber backfilled with helium the helium should be as high as possible consistent with keeping low frost temperatures. Maintaining the above parameters during the pumping operation provide the basis for the unexpected gain in producing the high vacuums achieved by this invention. These high vacuums are especially useful because the problems of removing large amounts of hydrogen is of particular concern because of the increasing need to evaluate the operation of small space propulsion units in ground based space chambers.

While the present invention has been described with particularity in reference to specific embodiments, thereof, it is to be understood that the disclosure of the present invention is for the purpose of illustration only and is not intended to limit the innvention in any way the scope of which is defined by the appended claims.

What is claimed is:

1. In a process for producing a high vacuum within a closed vessel having a gaseous atmosphere selected from the group consisting of hydrogen, helium and neon which comprises the steps of forming a cryodeposited sorbent gaseous frost structure on a cryogenically cooled surface and placing said gaseous atmosphere in communication with said sorbent frost structure to effect the cryosorption pumping of said sorbate, the improvement which comprises forming

said frost by depositing a sorbent gaseous material selected from the group consisting of carbon dioxide, sulfur dioxide, nitrous oxide, methylchloride, carbon tetrachloride, argon and nitrogen upon a surface cryogenically cooled to a temperature not in excess of 30° K at a pressure of at least 1×10^{-4}mm of mercury and at a deposition rate of at least 1.0 micron per minute to form a cryodeposited frost layer not in excess of one mm thick and maintaining said frost at a temperature not to exceed 25° K during said cryosorption pumping.

2. A process in accordance with claim **1** wherein sorbate atmosphere is hydrogen and said sorbent material is carbon dioxide deposited upon a surface cryogenically cooled to a temperature between about 12° K to 16° K.

3. A process in accordance with claim **1** wherein said sorbate atmosphere is helium and said sorbent material is carbon dioxide deposited upon a surface cryogenically cooled to a temperature of about 4.2° K.

* * * * *