

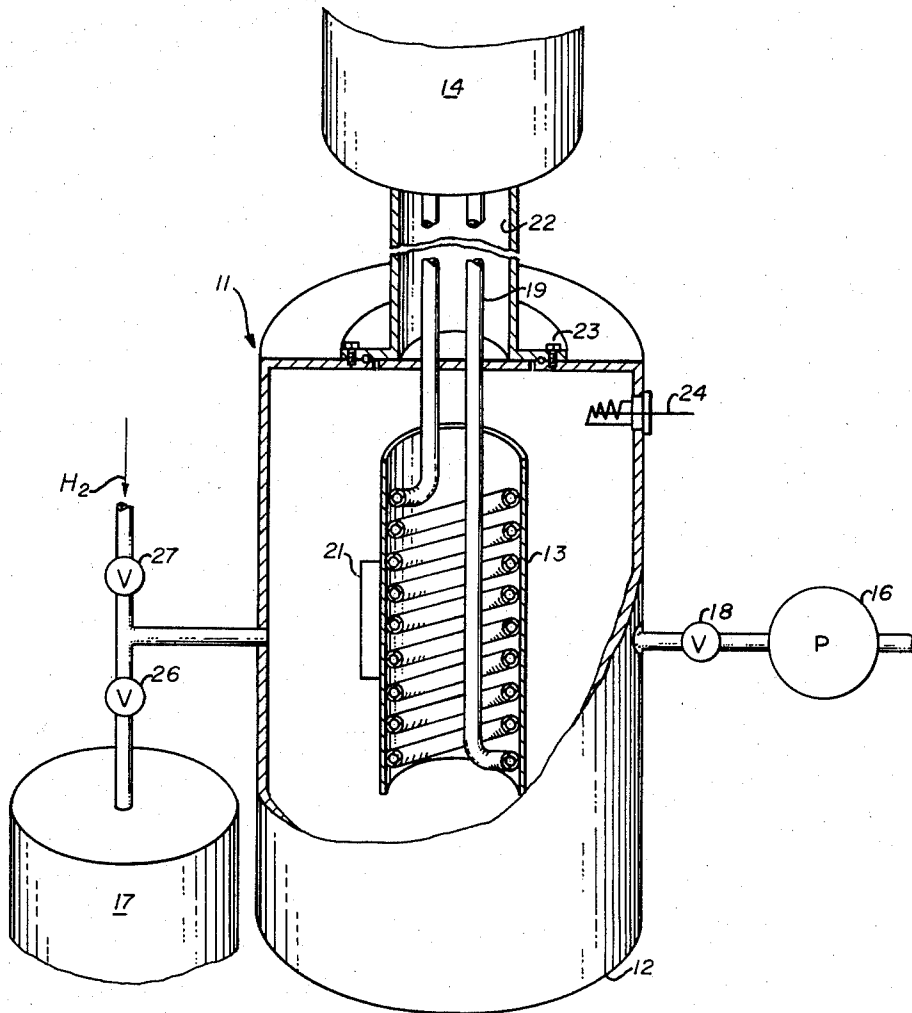
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PROCESS AND DEVICE FOR CRYOGENIC ADSORPTION PUMPING

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PROCESS AND DEVICE FOR CRYOGENIC ADSORPTION PUMPING

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The present invention relates to vacuum pumping and, more particularly, to a process and device capable of pumping hydrogen gas at a high rate. In general, the device comprises an adsorption element having a thermally conducting surface within a vacuum chamber, means for evacuating said chamber, to high vacuum—of the order of 10^{-7} mm. Hg, means for cooling the aforementioned surface to about 11° Kelvin and means for the alternate introduction of either hydrogen or a substrate gas into said chamber.

In the past, it has generally been considered necessary to bake large metal vacuum systems to at least 400° C. to reduce later degassing from the walls and to allow use of conventional pumping methods. However, there are several practical difficulties inherent to this high temperature treatment of these large metal vacuum systems, some of which are as follows:

A major difficulty is that if access to the interior is not to be severely restricted, demountable vacuum seals, commonly with metal or Teflon gaskets, must be used on the vacuum ports. Demountable seals often fail under high-temperature treatment. Further, thermal treatment of large structures requires very exact mechanical design due to the expansions and contractions of the structures resulting from the aforementioned thermal treatment.

A problem that pertains more specifically to the area of plasma research is the maintenance of ultra-high vacuum conditions in these large metal vacuum systems under an appreciable hydrogen or deuterium influx. Heretofore, attempts have been made to solve this particular problem through the prolonged operation of large and expensive diffusion pumps, but faster pumping rates are desired in the furtherance of the above-noted plasma research.

The present invention overcomes the above-mentioned difficulties in a unique manner, i.e., depositing a solidified layer of a substrate gas upon a cold surface located within an already evacuated chamber. The system is then capable of cryo-pumping hydrogen gas, in the ultra-high vacuum region, at high pumping speeds and in relatively large quantities by adsorption of the hydrogen gas on the solidified substrate surface. The worth of this system is more readily apparent when considering the fact that the equilibrium vapor pressure of hydrogen gas is approximately 2 mm. Hg at approximately 11° K., a fact that, prior to the present invention, precluded the cryo-pumping of hydrogen gas at high vacuum pressures.

In a preferred embodiment of the present invention, solidified gas films of A, N_2 , O_2 , H_2O , N_2O , and CO_2 have been individually used and hydrogen gas has been rapidly adsorbed at 11° Kelvin on all of them. This pumping of hydrogen was done while the chamber was evacuated to the 10^{-9} mm. Hg pressure range, and it is to be noted that the vapor pressures of all of the above gases at 11° Kelvin are less than 10^{-10} mm. Hg. Indeed, the use of any gas whose vapor pressure at 11° Kelvin is less than the degree of vacuum desired is within the scope of the present invention.

It is to be noted that a preferred embodiment of the present invention pumps hydrogen gas at a very high rate. However, reduced to its simplest form, the general param-

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eters of the present invention comprises; the equilibrium vapor pressure, at the cryogenic temperature desired, of the gas to be pumped, the environmental vacuum pressure required, and the equilibrium vapor pressure, again at the cryogenic temperature desired, of the substrate gas. More particularly, the equilibrium vapor pressure of the substrate gas should be less than the environmental vacuum pressure.

The high vacuum chamber associated with a preferred embodiment of the present invention is constructed of aluminum alloy; the flanges common to this chamber being sealed with Teflon rings. While in general such a construction in the past has limited base pressures to 10^{-7} mm. Hg, it is possible through the use of the present invention to obtain and maintain pressures of approximately 10^{-9} mm. Hg while pumping hydrogen at a speed of 60,000 liters per second.

It is a primary object of the present invention, therefore, to provide a cryogenic adsorption pump capable of pumping hydrogen gas.

It is a further object of the present invention to provide a process for pumping gas.

Other objects and advantages of the present invention will be more readily ascertained from an inspection of the following specification, taken in connection with the accompanying drawing, of which the single figure is an isometric, sectional view of the invention, while the features of novelty will be more distinctly pointed out in the appended claims. Referring to the drawing, there is shown a schematic presentation of a preferred embodiment of a cryogenic adsorption pump 11 comprising a vacuum chamber 12, an adsorption element 13 within said chamber, a helium refrigerator 14 for cooling element 13, conventional pump means 16 for evacuating said chamber, and a reservoir of substrate gas 17.

A preferred embodiment of pump 16 is an oil diffusion pump which is trapped with a liquid nitrogen trap of ultra-high vacuum design and is connected to the high vacuum chamber 12 through an all-metal valve 18 as is also shown schematically in the drawing. In this preferred embodiment helium coolant for adsorption element 13 is transferred from refrigerator 14 to the high vacuum chamber 12 through copper lines 19 which are insulated by a space which is evacuated by conventional means (not shown).

As shown in the drawing, a preferred embodiment of adsorption element 13 is a copper cylinder with an interior winding of transfer lines 19. Helium gas thermometer 21 monitors the temperature of this copper cylinder. Capillary tubing for this thermometer is brought out of this high vacuum chamber through the interior of transfer lines 19 to prevent any temperature transition regions which might establish high vapor pressure surfaces.

Although a copper cylinder is shown, it is to be understood that any object having a surface capable of being cooled to approximately 11° Kelvin would provide an adequate adsorption element. Indeed, the inner walls of vacuum chamber 12 could be used if adaptable to suitable cooling means.

The process of operating the above apparatus to achieve high speed pumping of hydrogen and deuterium gases is also novel. In order that the process be better understood, reference is once more made to the drawing.

To initiate a preferred operation, oil diffusion pump 16 is connected to high vacuum chamber 12 through all metal valve 18, as shown schematically in the figure. Chamber 12 is then pumped down to about 10^{-7} mm. Hg by means of pump 16.

To continue the preferred operation (above), the surface of adsorption element 13 is cooled to about 11° Kel-

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vin by helium refrigerator 14. The helium coolant for cooling element 13 is transferred from refrigerator 14 to vacuum chamber 12 by means of transfer lines 19. Cold transfer lines 19 enter high vacuum chamber 12 through flange 23 and are soldered to the interior of surface 13, thereby partially supporting said surface. As surface 13 is refrigerated to 11° Kelvin, the pressure range within chamber 12 decreases to the 10⁻⁹ mm. Hg range. This pressure is indicated by ionization gauge 24 which extends into the evacuated space through a seal means.

Then, the substrate gas is emitted from reservoir 17 through valve 26 and into chamber 12 where it forms a solidified gas film upon adsorption element 13. Various gases have been used with the pump, supra, and of these gases CO₂ and N₂O have been selected as preferred substrates since they absorb 40% of the hydrogen molecules falling on them until approximately 10¹² hydrogen molecules are absorbed per square centimeter of their surface. Because of the low vapor pressure of these preferred substrates at the low temperature of operation, the vacuum conditions within chamber 12 are not permanently impaired by their introduction. This is due to the virtually complete solidification of the substrate upon adsorption element 13. The system is now capable of pumping hydrogen gas at a high rate.

At this time valve 27 is opened and hydrogen gas is thereby admitted to chamber 12. As this gas is admitted, the rate of pumping gradually decreases due to the saturation of the solidified substrate surface. When the pumping speed has decreased below a desired rate, as is evidenced by a higher reading on ion gauge 24, the pump is reactivated.

Reactivation of the pump is initiated as follows; thermally conducting surface 13 is increased in temperature to the order of 15° Kelvin, i.e., between the hydrogen triple-point temperature and the normal boiling temperature. At this temperature the hydrogen gas vapor pressure increases to the extent that it is desorbed from adsorption element 13 and is slowly pumped out of chamber 12 by means of pump 16. The temperature of adsorption element 13 is again decreased to 11° Kelvin. This saturation and reactivation may be continued as a cycle without replacing the solidified substrate.

The above-mentioned steps, in combination, comprise a process whereby large quantities of hydrogen gas are pumped at high rates. Although hydrogen gas has been specified as the gas to be pumped, it will be apparent to those skilled in the art that many other gases with the notable exception of helium gas, can be likewise pumped through the use of the present process. This pumping of gases other than helium would merely entail an adjustment in parameters namely that of the substrate gas used.

While the present invention has been described in detail with respect to one embodiment thereof, it will be apparent that numerous modifications may be made within the spirit and scope of the invention. It is therefore not desired to limit the invention to the exact details shown except insofar as they are defined in the following claims.

What is claimed is:

1. In a cryogenic adsorption pump of the class described herein, gas adsorption means disposed in an evacuated chamber and comprising

- (a) a surface cooled to a temperature of approximately 11° K., and
- (b) a solidified layer of gas having a vapor pressure at approximately 11° K. less than the pressure of said evacuated chamber, said solidified layer being adhered to said surface.

2. In a cryogenic adsorption pump for hydrogen gas of the class described herein, hydrogen gas adsorption means disposed in an evacuated chamber and comprising

- (a) an adsorption element having a surface cooled to a temperature of approximately 11° K., and
- (b) a solidified layer of gas selected from the group

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consisting of gases whose vapor pressures at approximately 11° K. are less than approximately 10⁻¹⁰ mm. Hg, said solidified layer being adhered to said surface.

3. In a cryogenic adsorption pump of the class described herein, an adsorption element comprising

- (a) means defining a surface area,
- (b) refrigeration means, said refrigeration means being positioned in thermal communication with said surface so as to cool said surface to approximately 11° K., and

(c) a solidified layer of gas selected from the group consisting of gases whose vapor pressures at approximately 11° K. are less than the pressure of the environment within which the adsorption element resides, said solidified layer being adhered to said surface area.

4. The adsorption element according to claim 3 wherein said solidified layer of gas is selected from the group consisting of A, N₂, O₂, H₂O, N₂O, and CO₂.

5. The adsorption element according to claim 3 wherein said adsorption element comprises a copper cylinder.

6. A cryogenic adsorption pump for hydrogen gas comprising

- (a) a vacuum chamber,
- (b) conventional pump means in gas-tight communication with said vacuum chamber,
- (c) an adsorption element positioned within said vacuum chamber,
- (d) cooling means in thermal conduction relationship with said adsorption element,
- (e) a reservoir of gas, the gas in said reservoir having a vapor pressure, when solidified upon said adsorption element, which is less than the pressure established in said vacuum chamber, said reservoir being in gas-tight communication with said vacuum chamber, and
- (f) means for introducing hydrogen gas into said vacuum chamber.

7. The cryogenic adsorption pump according to claim 6 wherein the conventional pump means comprises a liquid nitrogen trapped oil diffusion pump of ultra-high vacuum design, said pump being in valved communication with said vacuum chamber.

8. The cryogenic adsorption pump according to claim 6 wherein the cooling means comprises a helium refrigerator, said helium refrigerator being in thermal conduction relationship with the aforementioned adsorption element by means of cryogenic fluid transfer lines.

9. The cryogenic adsorption pump according to claim 6 wherein the gas in said reservoir is selected from the group consisting of gases whose vapor pressures are less than 10⁻¹⁰ mm. Hg at approximately 11° K.

10. The cryogenic adsorption pump according to claim 6 wherein the gas in said reservoir is selected from the group consisting of N₂O and CO₂.

11. In a process for pumping gas, the step comprising adsorbing the gas upon a surface cooled to cryogenic temperatures and upon which is adhered a solidified layer of gas selected from the group consisting of gases whose vapor pressures are less than the background pressure of the surrounding environment.

12. In a process for pumping hydrogen gas, the steps comprising

- (a) introducing hydrogen gas into an evacuated chamber containing an adsorption element, the surface of which is cooled to 11° K. and upon which is adhered a solidified layer of gas selected from the group consisting of gases whose vapor pressures are less than 10⁻¹⁰ mm. Hg, and
- (b) adsorbing said hydrogen gas upon said adsorption element.

13. In a process for pumping hydrogen gas, the steps comprising

- (a) evacuating a vacuum chamber,
- (b) cooling a thermally conducting surface within said chamber,
- (c) introducing an amount of substrate gas into said

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chamber such that the gas solidifies on the thermally conducting surface, and

(d) adsorbing the hydrogen gas to be pumped on said solidified substrate.

14. In a process for pumping hydrogen gas, the steps 5 comprising

(a) evacuating a vacuum chamber to a low pressure of approximately 10^{-7} mm. Hg,

(b) cooling a thermally conducting surface within said chamber to a temperature of approximately 11° K., 10

(c) introducing an amount of substrate gas selected from the group consisting of gases whose vapor pressures are less than 10^{-10} mm. Hg at 11° K. into said chamber,

(d) solidifying said substrate gas upon the aforementioned thermally conducting surface, 15

(e) introducing the hydrogen gas into the aforementioned vacuum chamber, and

(f) adsorbing said hydrogen gas upon the solidified layer of substrate gas. 20

15. In a process for pumping hydrogen gas according to claim 14 wherein the substrate gas is selected from the group consisting of A, N₂, O₂, H₂O, N₂O, CO₂.

16. In a cyclic process for pumping hydrogen gas, the steps comprising 25

(a) evacuating a vacuum chamber to a low pressure of the order of 10^{-7} mm. Hg,

(b) cooling a thermal conducting surface within said chamber to a temperature of the order of 11° K.,

(c) introducing an amount of substrate gas selected 30 from the group consisting of gases whose vapor pressures are less than 10^{-10} mm. Hg at 11° K.,

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(d) solidification of said substrate gas on the aforementioned thermally conducting surface,

(e) introducing the hydrogen gas into the aforementioned vacuum chamber,

(f) adsorbing said hydrogen gas upon the solidified layer of substrate gas,

(g) heating said thermally conducting surface within said chamber to approximately 15° K.,

(h) desorbing said pumped hydrogen gas from said solidified layer of substrate gas,

(i) removing said desorbed hydrogen gas by conventional pump means from said vacuum chamber, and

(j) recooling said thermally conducting surface to approximately 11° K.

17. In a process for adsorbing hydrogen gas, the steps comprising:

(a) evacuating a vacuum chamber to a low pressure of approximately 10^{-7} mm. Hg,

(b) cooling a thermally conducting surface within said chamber to a temperature of approximately 11° K.,

(c) introducing an amount of substrate gas of the class of gases having a vapor pressure less than 10^{-10} mm. Hg at approximately 11° K. into said vacuum chamber to solidify upon said thermally conducting surface, and

(d) exposing said solidified substrate gas to the hydrogen gas which is to be adsorbed, thereby adsorbing said hydrogen gas.

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