ABSTRACT OF THE DISCLOSURE

Pumping systems for production of high vacuums frequently employ combinations of ionic and gettering pumping action and a trap which may be of the gettering or cryogenic type, but are limited by inert gas instability in the ion pump, which is avoided according to this invention by introducing a selected gas between the ion pump and a trap which is located between the ion pump and the high vacuum chamber, the gas being of a kind effectively removed by both the pump and the trap. Selected combinations of ion pumping, trapping or gettering, and introduced gas reduce the residual pressure to a point some orders of magnitude below the instability pressure at which argon, helium and other noble gases otherwise present cause interruption in the operation of the ion pump. By this invention one releases oxygen, hydrogen, etc., into the ion pump then ion pumps the mixture of noble gas and introduced gas to reduce residual pressures in combination with gettering or trapping the introduced gas in an improved trap.

According to various prior art vacuum production techniques a mechanical forepump or other pumping means reduces the pressure to $10^{-3}$ torr, more or less. A further high vacuum pump connects to the vacuum chamber to provide pressure in the order of $10^{-7}$ or $10^{-8}$ torr. The residual pressure may be further reduced by high temperature baking of the surfaces of the ion pump, or other high vacuum pumping apparatus, and the chamber to be evacuated, in order to remove much of the layer of gas molecules normally clinging to the surfaces, which molecules slowly escape from the surfaces to prevent very high vacuum results. This baking is termed "outgassing." However, bakeout is not fully effective to remove all pumped generated gas sources. Ion pumps do not operate to produce very high vacuums without gettering action to assist in removing the pump generated gas sources. Noble gases are not removed by absorbing or combining with the active metal surfaces produced by sputtering or subliming of an active metal within the ion pump. Ion pumps are ineffective for pumping inert gases when the residual pressure becomes so low that the electrical discharge involved in the ion-pumping action terminates for lack of sufficient gas molecules to produce repeated collisions for formation of ions to be swept out by the applied electrical field. The pressure at which ionic pumping terminates is somewhat variable but may occur at about $10^{-11}$ torr or higher.

High vacuums have been obtained by placing a getter and/or a cryogenic trap between the ion pump and the vacuum chamber. The trap serves as a pump for gases which react with an active metal evaporated upon the surfaces of the trap and thereby being bound by the active metal surface and removed from the system, or by condensation of high vapor pressure gases on the cryogenic surfaces. Such a trap is both a pump and a trap operating to prevent passage of molecules released from the ion pump and passing randomly back into the vacuum chamber. Refluxing preferably with liquid nitrogen to enhance the trapping effect with respect to gases which can be condensed at such temperatures except for certain well known capture reactions where elevated temperatures are preferable. A gettering or sublimation trap uses an active metal such as titanium sublimed from a point usually within the trap to the surfaces with which the passing gas molecules come into contact, which surfaces may be continually renewed by sputtering action during ionic pumping. Such a trap is not effective for noble gases since argon, helium, and neon are not easily condensed and the active metal surfaces do not react chemically with the noble gases. Since these gases are present in the atmosphere and are partially retained on the walls of the chamber, pump and trap, outgassing improves the resulting vacuum. However, when outgassed to achieve a better vacuum the problem of ion pump instability arises. Minute quantities of argon or helium normally present are then neither pumped nor trapped and the ion pump instability remains the limiting factor preventing the very high or ultra high vacuum conditions frequently desired. In essence, the pumping and trapping aspects of a cryogenic getter trap have been essential to very high vacuums but are not effective for noble gases at very low pump pressures because of the inert gas instability. For these reasons practical limits on combined ion-pumped and cryogenic getter-trapped well backed out systems have been between $10^{-11}$ and $10^{-13}$ torr.

It is accordingly an object of the present invention to provide apparatus and method for eliminating ion pump instability.

Another object of the invention is to shorten the time required for pumping a chamber to a high or very high vacuum.

A further object of the invention is to provide a pumping system having a high exhaustion rate for noble gases comparable to the exhaustion rate for the active gases.

A still further object of the invention is to provide a method and apparatus for practicing the method of deferring inert gas instability by the introduction of a gettering gas in small quantities within or ahead of the ion pump and behind the trapping trap.

These and other objects of the invention will be appreciated as the invention is described in connection with the drawings in which:

FIG. 1 is a schematic diagram of a pumping system according to one form of the invention;

FIG. 2 is a diagrammatic plot of pump speed versus pressure showing a region of argon instability.

FIG. 3 is a plot of pressure versus time in an ion-pumped system showing intermittent pumping action due to argon instability;

FIG. 4 is a plot of pressure versus time within the pump and vacuum chamber according to this invention; and

FIG. 5 is a detailed diagram of a preferred form of trap according to the invention.

Objectives of the present invention are achieved in pumping systems which have been referred to as getter-ion pumps, sputter-ion pumps, or orbi-ion pumps, in which electric field pumping action is combined with gettering action where the electric field pumping is maintained by intentional addition of a getterable gas, thereby enhancing the vacuum by several orders of magnitude of residual pressure reduction. As illustrated in FIG. 5, an active metal such as titanium is normally evaporated periodically to coat the surfaces of the trap 11 and pump 28, or may comprise the walls and baffle surfaces 14, 15 subject to particle bombardment to maintain freshly exposed active metal. Valves used for convenience in passages 7 and 16 are conventional and not shown.

According to one form of this invention a limited quantity of gas to a selected active type is introduced into the passage 7 by way of valve 8 and a constriction
9" in a tube 9 extending from a gas source 10. The particular form of the source 10, tube 9 and valve 8 selected is not critical nor is the location of its outlet either within passage 7 or within trap 11 or pump 3. However, a construction as at 9 is preferably employed when gas is introduced from a gaseous state source so that the rate of flow is restricted to permit only as much gas to be introduced as can be readily exhausted by pump 3 to approach the point of instability. This may be further adjusted by valve 8 or by other suitable means as may be desired.

Trap 11 is connected directly to the vacuum chamber either by a passage 16 or by bolting chamber 17 directly to the end of the trap 11. An improved trap according to this invention comprises one or more peripheral constriictions or baffles as at 13 and centrally located openings to passages 7 and 16 as illustrated in FIG. 1, which are effective to cause gas molecules moving by ballistic paths between impacts from pump 3 toward chamber 17 to strike walls or baffles 13 as they pass around baffles 12 which are preferably centrally located. It is preferable that at least two contacts on active surfaces are made by each molecule which may be moving randomly at thermal velocity from passage 7 toward chamber 17.

An alternative source of gas supplied at a limited rate is illustrated at 18, located in trap 11, comprising a small quantity of a solid hydrated salt, a hydrate, or certain selected oxides known to liberate oxygen at a desired rate at the effective operating temperature. For this purpose a solid substance is initially placed in the pump or trap passage and caused to decompose or evaporate at a predictable rate to furnish a supply of gas which is wholly internal to the system. It will be understood that the gas selected for controlled release for this purpose must be selected from various known slowly gassing elements or compounds which will not be excessively depleted by the baking out process. Furthermore, the gas source selected must one which is readily pumped or trapped by both the ion pump and the selected trap or getter. Gas such as oxides of carbon or water vapor may be introduced by inclusion in trap 11 or passage 7 as a solid hydrated salt or hydrate, which is then caused to liberate the desired gas.

As an example, mercury vapor may be introduced by way of valve 8 and is effectively pumped at 3 to greatly reduce the residual inert gas pressure without itself contributing to the residual pressure. As shown in FIG. 17 since it is trapped chemically or physically in 11.

It may be noted that water vapor and certain hydrocarbons are readily trapped either by cryogenically cooled surfaces or active metal surfaces, both of which are illustratively combined in trap 11. Any gas may be selected which is readily trapped or gettered by active metal surfaces even though it is not condensable upon cryogenically cooled surfaces, as in the case of hydrogen or oxygen. In the case of certain gases present or introduced it may be desirable to avoid the use of a cryogenic cooling of the baffles or walls in trap 11 since elevated temperatures are more effective, as in the case of chemical combining of oxygen with certain metals.

In FIG. 3 is shown the effect of instability in an ion pump, commonly referred to as "argon instability." As pumping proceeds, the pressure in the ion pump reduces to a point such as 20 on curve A at which time ionization becomes insufficient to maintain a discharge. Whenever the pumping action caused by ionization products being swept forward by the electrical fields terminates the pressure would remain constant except for the continuously occurring release of molecules adhering to the walls of the pump passages communicating with the pump. Argon is particularly troublesome because of its substantial concentration in the atmosphere to approximately one percent and its tendency to be released from the surfaces of the ion pump. Thus when ion pumping reaches a point as at 20 on curve A no further crying pumping of any gas occurs at pump 3, but combinable gases would be pumped to very low pressures by trap 11 and active surfaces in pump 3. Since the inert or noble gases are not removed by this gettering action on the active metal surfaces of either the ion pump or the trap a slow rise of pressure by which the rate of flow is restricted to permit only as much gas to be introduced as can be readily exhausted by pump 3 to approach the point of instability. This may be further adjusted by valve 8 or by other suitable means as may be desired.

In FIG. 4 curve C shows the result of introduction of gas into the ion pump or into the trap or passage adjacent to the pump. When a trapable gas is introduced into the pump at a rate which permits a maximum or near maximum pumping rate, it will be understood that the noble gases present in the chamber and the trap will continue to be pumped along with the trapable gas without the pumping interruptions shown in FIGS. 2 and 3 caused by too low a pressure in pump 3. In addition the pumping rate remains higher as shown in FIG. 2 at somewhat higher pressure levels. Thus the introduction of any gas at a rate sufficient to hold pressure above the instability point causes pump 3 to remain effective at all times. The instability point generally may be in the range of 10^-11 torr and is illustrated at E of FIG. 4. Resultant pressure in the ion pump after introduction of the trapable gas is kept above curve E and is shown by curve C. The result of continuing rapid pumping at 3 where the total pressure remains above curve E exhausts argon, helium, etc. by ion pumping action. The remaining argon, helium, etc. is reduced continually since its pumping rate is maintained. It will be seen that this is equivalent to extending curve B of FIG. 3 as shown at B', and the result is shown generally at D in FIG. 4.

While the effectiveness of cryosorption trapping of a condensable gas, or of a gettering gas active with respect to the active metal, will vary from system to system and in accordance with the adhered contaminating gases present after baking, high vacuums of the prior art are subject to very large improvements into the ultra high vacuum range by this method. It will be seen to be somewhat like the liquid nitrogen trapping of a mercury vapor pump. A ratio of 10^-6 between the final pressure of 10^-4 torr and 10^-2 torr vapor pressure for mercury at atmospheric temperatures is experienced by mercury vapor trapping in conventional practice. Cryosorption trapping and gettering of the introduced gas leads to final pressure values in the order of 10^-9 torr or better.

Referring to FIG. 5 it will be noted that a pump is generally indicated at 25 connecting to passage 7 and having a source of gas introduced therein at 29. This pump may be of the form shown in FIG. 1 at 3 or may operate on any other well known principle effective to remove gases encountered to some limiting instability pressure such as that experienced with the ion pump. Introduction of gas by the tube 29 (or as shown at 18 in FIG. 1) and continued pumping of the mixture of introduced and residual gases effectively dilutes the inert gases which would be present in trap 11 and passage 16, hereinafore described with particular reference to argon or helium. When these gases are introduced in a totally removable by trap 11 it will be seen that pump 28 operates to reduce the partial pressures comprising the residual pressure in the chamber as fast as they are released and at chamber pressures considerably lower than the instability of pump 28. The net effective pressures which can be obtained in this manner will be the product of the controlled operating pressure of pump 28, above the unstable operating region, and the trap effectiveness for removing the introduced gas. For example if pump 28 is operated at 10^-10 torr by injection of a gas for which
3,502,259

5 trap 11 has a pressure ratio of $10^{-5}$ then pressure of $10^{-16}$ torr can be achieved. In FIG. 5 a particular form of trap enclosure 11 is shown in which active metal surfaces 14 and 15 cover walls and baffles of the trap. Baffles 13 are combined with inner walls 22, and additional baffles 24 and 25 to cause randomly moving gas molecules from passage 7 to strike at least two surfaces of the trap before they can reach passage 16. Diaphragm 23 is shown illustratively as supporting walls 22 and baffles 13, 24 and 25 from which baffles 12 may also be supported in a manner not shown. The form of trap illustrated in FIG. 5 is adapted for cryogenically cooled surfaces to be cooled by liquid nitrogen. The like, admitted thereto by conduit 26 from a source 27. Diaphragm 23 is used to block off any gas passing between the trap wall and the interior cryogenically cooled portions containing active metal deposits to ensure that all gaseous molecules from pump 28 are forced to collide with active cooled surfaces.

We note that in any of the getter-electric field types of pumps, if the discharge is maintained by secondary electron emission (as opposed to electron emission from a hot filament) the ionizing pumping portion will extinguish at some value of pressure. At this point, the gettering action will cease. In the Evapor-Ion pump, active metal is constantly supplied by evaporation so getter action never dies. In sputter-ion pumps, the sputtering of the surface depends on the ionization discharge. Hence, if the discharge is extinguished, no new supply of active metal is available. Under this condition, the gettering speed will begin to decrease as the available surface material is used up. If no inert gas is available to cause the pressure to rise and reinitiate the discharge (which in turn reactivates the gettering surface), then eventually the gettering action decreases to the point where the partial pressure of active gases begins to rise. This will reinitiate the electronic discharge. We note then, that not only is there an "argon instability" or "inert gas instability," but also an active gas instability if the pump is clean and no inert gases are present. While in most systems there is sufficient argon buried in the active metal surface of the pump so that the argon outgassing controls the reinitiation of the discharge, the present invention serves to avoid both argon instability and active gas instability.

From the foregoing description it will be seen that ultra high vacuums may be produced by the intentional introduction of extraneous gas into the vacuum system, which gas is of the kind removed in an improved trap of high efficiency, and that the improvement achieved is directly related to the trapping ability of the high efficiency trap for the introduced gas and the pump instability pressure. Applied to the input end of an ion pump the gas supply and trap combination eliminates argon instability to provide a higher order of vacuum than is otherwise obtained. The improvement ratio is related closely to the ratio of introduced to residual gas when the trapping is made wholly effective by the selection of a trapable gas which cannot pass backwards through two or more impacts with the active metal trap surfaces. Thus the invention is principally applicable to elimination of the limitations inherent in pumping by electric discharge. Such pumps may be considered to operate upon the mixture of gases present to provide a residual total pressure of a magnitude according to efficiency of the pump.

It will be understood that the invention is not limited to the specific form of high vacuum pump or to a particular configuration of trap, but is applicable to a number of combinations of these elements for gases known in the prior art to be trapable on active metal surfaces or to the trapped or cryogenic removal of introduced gases from surfaces. The pressures at temperatures prevailing on cryogenically cooled surfaces of the trap. Applicant accordingly does not wish to be limited to the specific steps and apparatus shown other than as indicated by the scope of the appended claims.

What is claimed is:

1. In the art of ionically pumping gases exhibiting argon instability from an enclosure to produce a high vacuum by apparatus having an unstable pumping rate at a point near its maximum pressure and removing partial pressures due to trapable portions of remaining gases in a trap having active metal surfaces, the improvement which comprises:

introducing a quantity of trapable gas in the vicinity of said surfaces sufficient to maintain the operating pressure above said minimum, and

removing said introduced gas by trapping at said surfaces, whereby non-trapped gases are reduced by continued trapping beyond the time of instability in the absence of the introduced gas.

2. In the art improvement of claim 1, said pump being an ion pump in which said instability arises from extinguishing of electrical discharge at said point, and said non-trapped gases being noble gases.

3. In the art improvement of claim 1, said steps of trapping including subjecting at least two said active metal surfaces impacted by molecules moving toward the enclosure to cryogenic cooling.

4. In the art improvement of claim 1, said step of introducing trapable gas comprising releasing over an extended pump period a gas contained in solid form initially placed in the vicinity of said surfaces.

5. In the art improvement of claim 1 said step of introducing trapable gas comprising passing said gas through a constricted at a controlled rate proportioned to pumping rate to maintain the operating pressure slightly above instability.

6. In the art of producing a high vacuum in an enclosure by ionic pumping and by trapping portions of residual gases upon surfaces of active metal with respect to the trapped gases, the method of removing untrapable gases comprising the steps of:

introducing a highly trapable gas into the region ionically pumped at a rate to maintain ionic discharge and pumping of untrapable gases.

ionically pumping the mixed trapable and untrapable gases, and

substantially removing said introduced gas by trapping to effect a reduced residual partial pressure of untrapable gas dependent upon the ratio of time for pumping to instability without introduced gas and the duration of ion pumping with introduced gas.

7. In the method of claim 6, said introduced gas being selected from the group of gases having a high known ratio of vapor pressure at ambient temperature to the vapor pressure at a selected cryostatic temperature and said trapping step including subjecting said active metal surfaces to said cryostatic temperature.

8. In the method of claim 6, said introduced gas being of the group comprising oxygen, hydrogen, nitrogen and the carbon oxides.

9. In the method of claim 8, said active metal being titanium.

10. In the method of claim 8 said trapping being effected by sputtering of a known active metal at surfaces of passages connecting to said region.

11. In the art of producing a high vacuum in a region containing noble gases tending to produce argon instability when ionically pumped and lowering residual partial pressures by gettering action, the method of the steps of introducing a readily gettable gas at a rate sufficient to maintain stable ionic pumping, and substantially exhausting said introduced gas by gettering action, to thereby continue the ion pumping of noble gases below the pressure below which instability occurs in the absence of the introduced gas.

12. In the art improvement of claim 11, said gettering action comprising sputtering of a metal active with re-
3,502,259

spect to said introduced gas and said gas being introduced at a rate greatly in excess of the rate of release of noble gases in said enclosure.

13. In the art improvement of claim 12, said introduced gas being introduced by decomposition of a solid containing said gas at a controlled rate sufficient to maintain total gas pressure ionically pumped above that pressure at which instability commences.

14. In an apparatus for evacuating an enclosure by ionic gas pumping and trapping in an active metal trap connected between said enclosure and said pump to produce an enhanced vacuum by substantial removal of partial pressures due to trapable gases contained, an apparatus for substantially reducing residual partial pressures due to non-trapable gases contained, comprising:
   a source of a trapable gas,
   means releasing said gas into the region of said trap and pump, and
   means for controlling the rate of release to maintain a combined pressure of said trapable and said non-trapable gases within the continuous pumping range for said gas pump.

15. In apparatus according to claim 14, said source of gas being a source of oxygen supplied at a rate approximating ionic pumping rate before argon instability commences.

16. In apparatus according to claim 14, said source of gas being a hydrated salt.

17. In an apparatus according to claim 15, said trap including cryogenically cooled surfaces for enhancement of gas entrapment.

18. In apparatus according to claim 14, said trap comprising baffled passages between said enclosure and said pump disposed to cause gas molecules to impact at least two surfaces in passing from said pump to said enclosure.

19. In apparatus according to claim 18, means cryostatically cooling portions of said trap to enhance capture thereon of molecules of trapable gas.

20. In apparatus according to claim 18, said source of trapable gas being oxygen and said baffled passages including means for maintaining temperature thereof above ambient.

21. In apparatus according to claim 14, said means of controlling the rate of release of trapable gas including a restricted conduit connecting said source to said gas apparatus ahead of said pump and substantially behind said trap.

References Cited

UNITED STATES PATENTS

3,140,820  7/1964 Clasus ----------------- 230—69
3,144,200  8/1964 Taylor et al. ------------ 230—69
3,155,310  11/1964 Lorenz ----------------- 230—69
3,164,320  1/1965 Welbourn ----------------- 230—69
3,252,652  5/1966 Trendelenburg et al. ... 230—69

ROBERT M. WALKER, Primary Examiner