Feb. 28, 1961 M. VON ARDENNE 2,973,134

ION GETTER PUMP

Filed Dec. 10, 1958 3 Sheets-Sheet 1

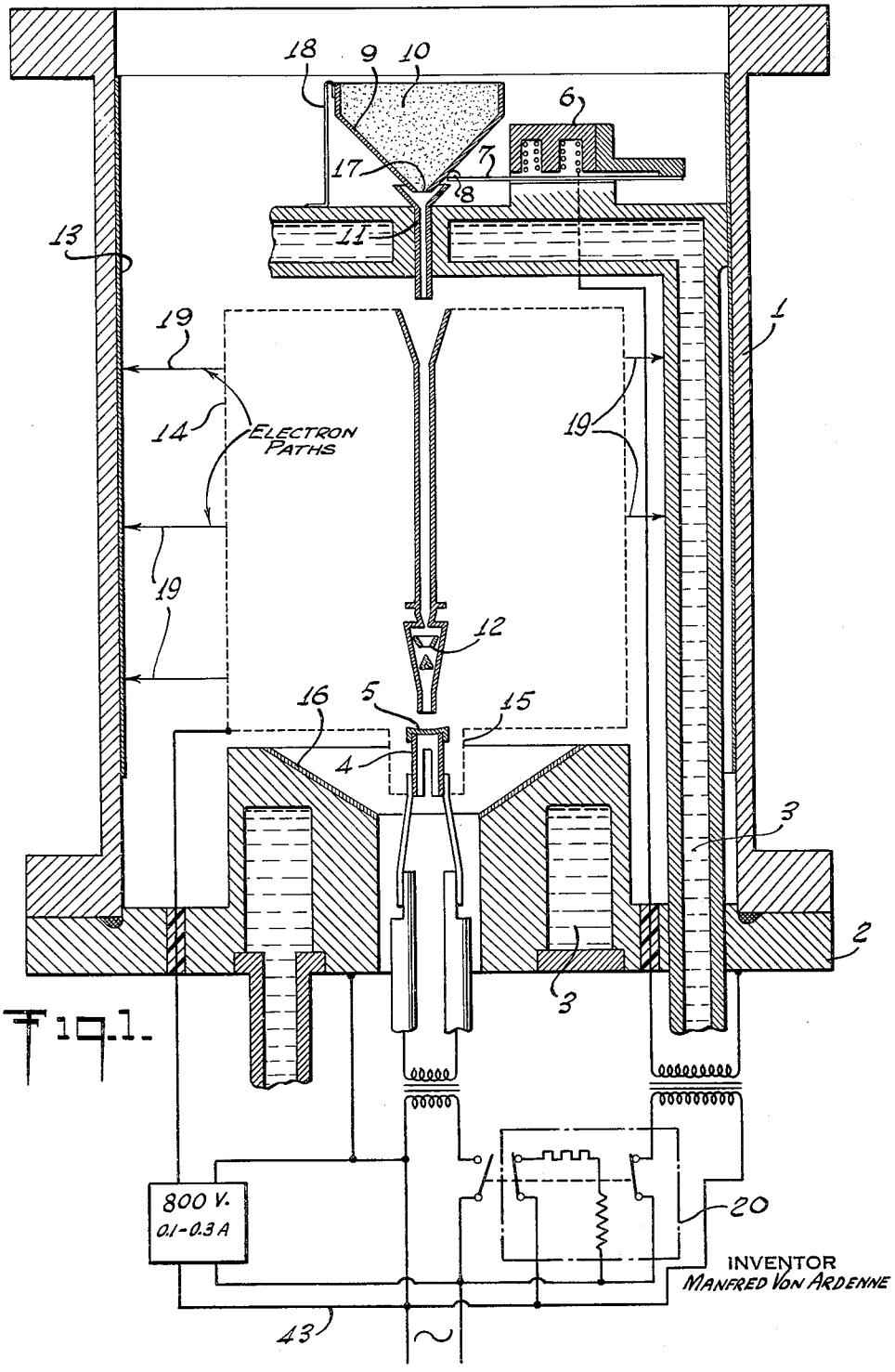


Fig. 1.

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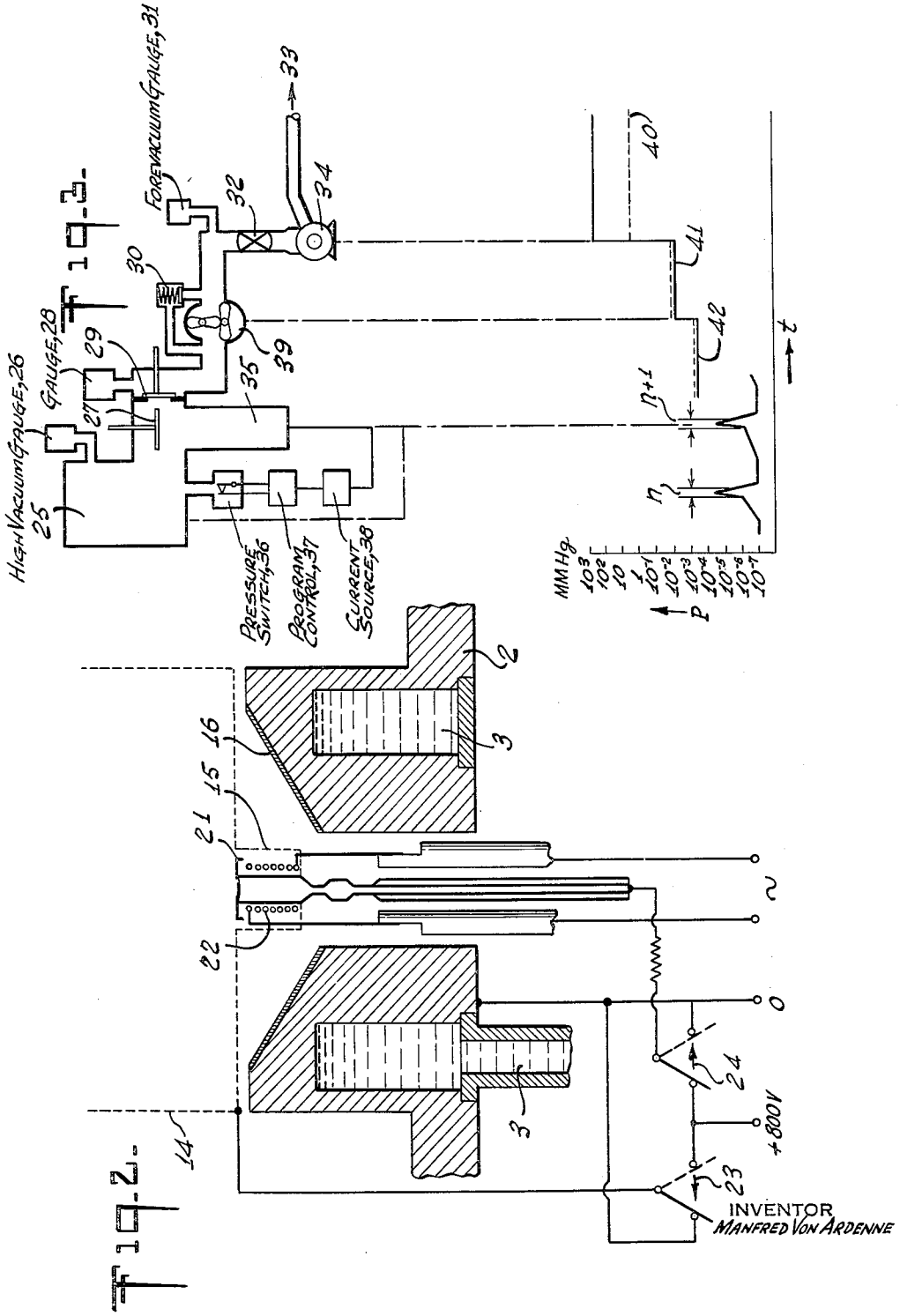

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Fig. 5.

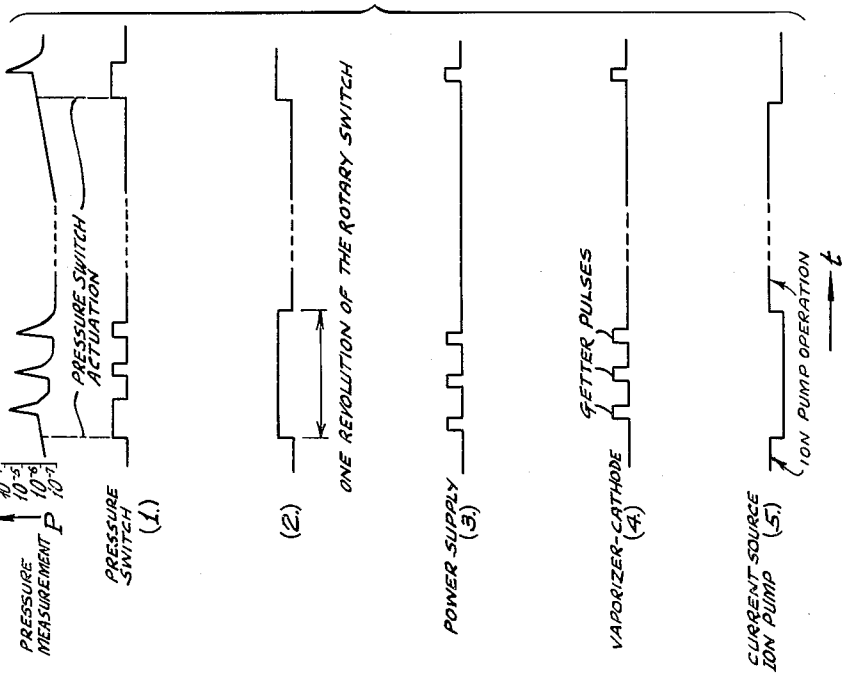
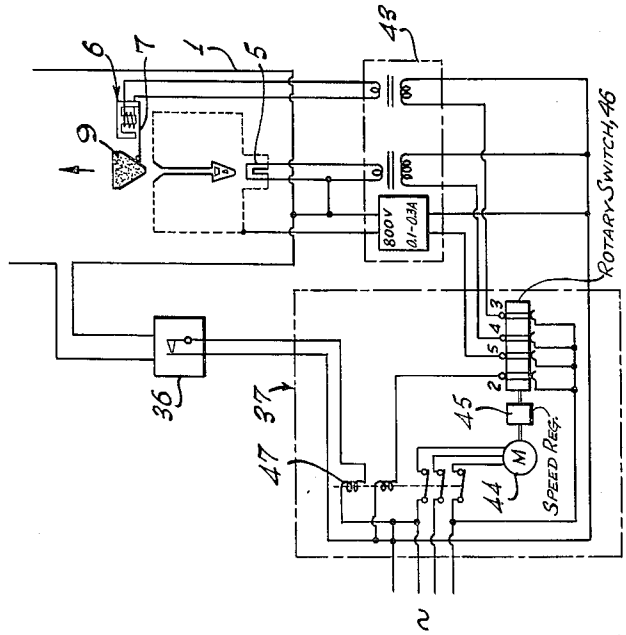



Fig. 4.




INVENTOR
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United States Patent Office 2,973,134 Patented Feb. 28, 1961



2,973,134

# ION GETTER PUMP

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Filed Dec. 10, 1958, Ser. No. 779,446

Claims priority, application Germany Dec. 12, 1957

12 Claims. (Cl. 230—69)

The invention pertains to improvements in an ion getter pump.

Ion getter pumps so far have not been used widely. The reason for this lack of practical application may be found in one or several disadvantages inherent in the designs of the pumps of this type which have become known until now.

The evaporators used for dispersing the getter metal are usually heated by electron bombardment to the high temperatures required. As a consequence, such a pump will start operating or, if started, will maintain stable operation only if the pressure of residual gas is first reduced to $10^{-4}$ mm. Hg or less.

According to another known type of ion getter pump design, the evaporator is heated by gas discharge which changes to a vapor discharge in the vapor of the getter metal as soon as evaporation of the getter material is initiated. This device requires the getter metal to be evaporated continuously without interruption in order to prevent a break in the discharge. As a result, the consumption of getter metal is higher by one or two orders of magnitude than would be necessary for merely producing the getter pump effect.

My copending patent application Serial No. 750,255, filed July 22, 1958, describes an ionic getter-pump, in which an electrode made of high-temperature melting material and heated by joulean heat is simultaneously employed as a getter-metal vaporizer and as a cathode for the ionic pump action. The getter-metal vaporizer consists, for example, of a cylinder made of tungsten sheet, through which the heating current flows and which simultaneously serves as a cathode for the pump action and as a vaporizing pan.

One advantage of the embodiment described in the above-mentioned application is that the employment of an axial magnetic field and of heating with alternating current keeps the electrodynamic forces acting on the cathode small, thus preventing any distortions or premature destruction due to vibration. Another advantage of the vaporizer is that the surface areas serving as the cathode are not touched directly by the getter metal and do not have their emissivity diminished or changed as the result of alloy formation, while, on the other hand, the cover plate serving for metal vaporization is hardly, or not at all, used for electron emission. The same voltage source draws off the electrons for the ionic pump action and at the same time accelerates the ions formed in the ionization chamber in the direction of the getter condensate layer. Auxiliary electrodes at approximately the cathode's process potential are provided to deflect the electron paths in the ionization chamber.

The invention of my prior application still possesses the deficiency, namely, that during operation difficulties may occur with respect to the purely chronological sequence of phenomena, due to unprogrammed, though possible modulation.

These disadvantages are eliminated in the invention by using a pressure switch to interrupt the heating of the



vaporizer cathode system automatically and chronologically after measurement of the pressure, during the brief supply of getter-metal powder, such as titanium-metal powder, thus preventing partial sintering of the getter-metal powder, whereas after vaporization is completed in the form of preferably several vaporization pulses, the current source is connected with the grid electrode until the required pressure values are achieved in such a way that the ionic pump action is superimposed on the already accomplished vaporization getter action and the initiated contact getter action.

An embodiment of the invention is described in detail, with reference to the drawings, wherein:

Fig. 1 is a view partly in section of the ion getter pump;

Fig. 2 is a partial view of the ionization producting means;

Fig. 3 is a schematic diagram of an assembly according to the invention, including means for producing a high pre-vacuum;

Fig. 4 is a schematic diagram showing the sequence controlling mechanism, and

Fig. 5 shows graphs which are explanatory of the operation of the system.

The mode of operation of the system will first be explained with respect to its getter-pump action. In Fig. 1 the pump system is shown in a metal housing 1, which is connected through a main vacuum valve 27 with a suitable opening to the receiver 25 (see Fig. 3). To produce the initial forevacuum, this valve is opened and the receiver and pump are jointly evacuated to the required pressure of about $10^{-4}$ mm. Hg. This type of connection makes it possible to maintain the ionic pump under vacuum when the main apparatus is shut down. In this way we are able to diminish substantially the absorption of gas by the getter-metal supply and the getter-metal layers, which otherwise diminish the suction velocity of the pump appreciably at starting.

The getter metal employed is a titanium metal powder 10, degassed by a process described below, in particular, freed to a large extent of its hydrogen content. The metal is used in its powder form because supplying the metal to the evaporator in powder form is easier than in the usual wire shape, and because the powder metal is more readily available and cheaper than the wire. The titanium metal powder 10 is poured into a thin-walled hopper funnel 9 suspended, say, by springs, which has a fine opening 17 at the bottom. The mean grain size of the powder and the size of the opening 17 in the hopper funnel 9 are of such dimensions that no powder falls out of the hopper when at rest. Only when an energizing magnet 6 is actuated and when the vibrator hammer 8 with vibrator tongue 7 attached to the hopper funnel 9 and set vibrating electromagnetically at, say, 50 cycles, strikes the funnel, does a fairly uniform flow of titanium metal powder particles 10 flow out of the hopper funnel 9.

The powder falls through a series of funnels and tubes 11 into a baffle 12, where it is slowed down. From this baffle it falls from a very low height on the vaporizing pan 5. Part 2 is the bottom flange of the housing, 3 is the water cooling arrangement, 13 is the metal sheet that received the titanium vapor, 18 the spring suspension of the hopper funnel 9, and 20 a delay relay.

The vaporizing pan 5 is not heated by electron impact, as in the industrial getter-ion pumps known hitherto, but by radiation and conduction of heat from a tungsten cathode 4 heated by joulean heat. One of the principal features of the arrangement of the invention is the fact that this same tungsten cathode 4 simultaneously supplies the electron emission for the ionic pump system.

The mode of operation of the system insofar as its

ionic pump action is concerned is described below. The electrons used for ionizing the residual gas, which are indicated by 19 in Fig. 1, are drawn off from the external surface of the tungsten cathode 4 by the collector section 15 of the grid 14, maintained at a potential of about +800 volts by source 43, this section being only a short distance away from the cathode, and then reflected into the ionization chamber proper just in front of an inclined surface, the electron mirror 16, which is at the cathode potentital (potential of the housing).

The ionization chamber is encased by a fine-wire grid 14, which is conductively connected to the collector section 15 of the plate grid mentioned above. The size of the outer surface of the tungsten cathode 4 and the collector distance are so dimensioned that at a collector voltage of about +800 volts and a high vacuum of, say, $p$=about $10^{-5}$ mm. Hg, a total electron current of about 0.1 to 0.3 ampere is deflected into the ionization chamber. By designing the grid 14 so as to have as little surface as possible and by avoiding the use of units of large area to transfer the powder into the interior of the ionization chamber we succeed in having the electrons that are reflected back into the ionization chamber after they pass through the grid meshes by the outer reflecting field possess a high oscillation factor. The high total current and the great length of the electron paths in the ionization chamber due to the high oscillation factor yield high efficiency in ionizing the residual gas. As soon as the ions that are formed pass through the grid 14, they are accelerated in the direction of the pump housing and shot into the titanium getter layer deposited there, which is periodically renewed.

Hence, aside from the slight energy required to energize the energizing magnet 6, the getter-ion pump of the invention requires only the electric power for heating the tungsten heater 4, and a source of current for 800 volts, 0.3 ampere.

A major advantage of the design of the vaporizer system of the invention, that is, dispensing with the hitherto customary heating by electron impact, consists in the fact that the pressure of the forevacuum required is higher and that in the event of sudden eruptions of gas the operation of the vaporizer heating remains stable and cannot be changed or put out of operation by the formation of plasma.

The getter metal proposed is, for example, titanium metal powder, which is degassed for 10 minutes at a temperature of approximately 1150° C. After degassing the getter metal powder, which has fritted together, is repulverized and sealed into dry ampoules in precisely the quantity corresponding to one filling of the hopper. In the getter-ion pumps described by way of example this quantity is about 20 grams. About 1.5 mg. thereof is vaporized during each vaporization pulse. This supply is enough for about 13000 vaporization pulses and under normal conditions is sufficient for more than 100 evacuations. The preliminary degassing frees the powder from most of its hydrogen, which represents the principal impurity, as well as of much of the originally adsorbed gaseous constituents ($O_2$, $N_2$, $CO_2$). Hydrogen is given off at a temperature of some 800° C.

The advisability of preliminary degassing is shown by the estimate that the original gas content is of the order of magnitude of 1 liter per mg. of titanium powder at $10^{-3}$ mm. Hg. By way of comparison it might be mentioned that about 100 times this quantity can be absorbed by titanium if the temperature of the titanium surface is chosen appropriately. Furthermore, the rate of getter action is particularly high for $O_2$ in the temperature range of 700° C., and for $N_2$ and $CO_2$ in the temperature range around 1000° C., while $H_2$ getter action occurs at a high rate even at room temperature. The foregoing figures apply to compact getter metal.

In dimensioning the pump system high temperatures are not chosen for the principal getter-metal collector surfaces, as the rate of collection is practically limited by the cross-section of the pump connection rather than by insufficient getter action.

The physical properties of titanium that are important to dimensioning of the vaporizer system are summarized below in tabular form.

Some properties of titanium:

Atomic weight A=47.9
Density=4.43
Melting point $F_p$=1668° C.

Saturation pressures:

$P_s=10^{-4}$ mm. Hg at 1440° C.
$10^{-3}$ mm. Hg at 1600° C.
$10^{-2}$ mm. Hg at 1755° C.
$10^{-1}$ mm. Hg at 1940° C.
1 mm. Hg at 2200° C.
10 mm. Hg at 2500° C.

The equation giving the specific rate of vaporization is:

$$a=5.85\cdot10^{-2}p_{s\,[\text{mm. Hg}]}\sqrt{\frac{M}{T_{[\text{oK}]}}}[\text{g. cm.}^{-2}\cdot s^{-1}]$$

In this equation:

$p_s$=Saturation pressure at the metal temperature T
$M$=Mass number≈atomic weight A

If the vaporizer is dimensioned so as to possess an effective vaporization area of about 0.2 cm.$^2$ and if we require that the 1.5 mg. of titanium metal powder fed in per pulse be vaporized in about 1 second, the figures in the table and the foregoing equation for the specific rate of vaporization indicate that the vaporization temperature is about 2200° C. The saturation pressure at the surface of the metal is then 1 mm Hg.

A disadvantage of the invention in patent application Serial No. 750,255 is the fact that the supply of powder through a thin tube that starts directly beneath the hopper opening is inadequate by itself. Falling through a distance of more than 10 cm, the powder bounces off the vaporization pan. That is why a baffle is installed at the end of the supply tube to brake the speed of fall of the powder. But in continuous operation, the intense radiation from the adjacent vaporizer very quickly obstructs the bottom opening of the baffle as the result of partial sintering of the powder falling through. The adherence of the powder to the wall of the tube is prevented by a wire that acts as a stirrer.

This disadvantage is eliminated in the present invention by operating the getter-ion pump intermittently. Intermittent operation is described in detail below. Another important reason for employing intermittent vaporization is the savings effected in titanium consumption.

The titanium powder 10, Fig. 1, to be vaporized falls into a recess, say, cup-shaped, in the vaporizing pan 5, made of, for example, fine-grained graphite of high density or of tungsten carbide. The vaporizing pan 5 is mounted on the upper end of a cylinder of sheet tungsten 4, heated by the direct flow of current through it. To secure a high temperature at the upper end of the cylinder, which is not lowered by heat conduction, and to diminish the current required for heating the cylinder is slotted, for example, nearly up to its top end. The portion of the cylinder's surface that contributes to electron emission is about 1.1 cm.$^2$. As ionization of the residual gas in the ionization system requires an electron current of no more than 0.3 ampere, a temperature of 2200° C. for the tungsten cathode 4 is adequate for electron emission, that is, for prolonged operation of the ionization system. The power required for heating is then 510 watts. To secure long service life of the heater with the lowest possible heater temperature during the vaporization period the vaporization pan 5 must be designed in such a way and combined with the tungsten cathode 4 in such a way as to have as small a temperature gradient as possible between the heater temperature and the

getter-metal vaporizing surface. It is also necessary that the vaporizing pan of graphite be shaped in such a way that the boundary zone of the pan is automatically at a temperature below the melting point of titanium.

This makes it possible to prevent liquid titanium from creeping over the edge of the pan to reach the heater, thus avoiding destruction of the heater by alloy formation. The hot inner zone of the heater is heated by thermal conduction over a short path as well as by black-body radiation to the bottom side of the pan from the interior of the tungsten cathode. When the cup-like design of the pan that diminishes the temperature gradient is employed, the temperature gradient between the titanium vaporization surface and the heater is about 300° C. Hence, the temperature of the heater must be raised to about 2500° C. to secure the rapid vaporization of the quantity of titanium supplied per pulse described above, which requires a temperature of 2200° C. As this higher temperature is required only briefly during vaporization proper, its use does not involve any critical shortening of the service life of the heater.

The getter collector surface is practically uniformly metal-coated over nearly the entire cylindrical inner wall of the pump housing notwithstanding the cupshaped recess in the vaporizing pan. The reason for this is that the mean free path of the vapor particles in the space directly above the cup-shaped recess during vaporization is less than 1 mm., so that only volumes at some distance from the recess become emission regions for atomic titanium rays. The recess, which is cup-shaped or funnel-shaped, for example, has the further advantage that it does a better job of keeping the falling powder together.

Starting at the edge of the cup, the graphite pan is only, say, 0.3 mm. thick. Hence heat transfer through conduction to the outer peripheral zone of the pan is low. Moreover this outer peripheral zone is heated only slightly by radiation. As a result it actually remains so cool that no liquid titanium can flow over the edge and destroy the heater.

Another feature is the manufacture of the evaporation pan of extremely fine-grained graphite in vacuum and under high pressure. Vaporizing pans produced by this process have a structure of maximum density, are completely free from cracks, and possess the desired shape. Tests with standard brands of graphite (volume of voids>20%) have shown that it is possible for the liquid titanium to leak through gradually to the bottom surface of the pan. This results in destruction of the heater through alloy formation after about 10 to 20 vaporization pulses. The finest hair-cracks have the same effect.

The above-mentioned difficulties do not occur when the vaporizer is heated by electron impact. But since it was often found that heating became unstable in this case, even at initial pressures of the order of $10^{-4}$ mm. Hg, and even a gaseous discharge between the vaporizer and the heating coil, it seems desirable not to employ the method of heating by electron current.

A system used, by way of example in the invention is shown in Fig. 2 together with its circuit.

Here part **21** represents the tungsten vaporizer, and part **22** the doubly utilized tungsten cathode. The position of the switch for action as an ionic pump is shown at **23**, and the position of the switch for action as a getter pump is shown at **24**.

The getter action of titanium for neutral atoms of rare gases, is slight, though it is perceptible for argon. Ions of the rare gases that are shot at the collector are held fast with a satisfactory degree of efficiency, however. With an accelerating voltage of 800 volts about every fourth argon ion remains in the Ti layer. That is why the vaporizing system described above is supplemented by an ionization system acting as an ion pump for getter action. The ionization system consists, as described above, of the hot cathode, the collector grid, the electron mirror, and the grid. The collector or accelerating grid for the electrons is conductively connected to the grid encasing the ionization chamber. This yields a much simpler design and operation of the pump. There is a potential difference of, say, +800 volts between the grid and the pump housing, in order to secure the above-mentioned 25% probability of capture for the argon ions drawn off the ionization chamber. The electrons emitted from the cathode, which is at the potential of the housing, are likewise accelerated with, say, 800 volts. To be sure, this exceeds the optimum electron velocity for ionization, but the ionization cross section drops only to about 40% of the optimum figure. To some extent, in a discharge of comparatively high current density the diminution of the ionization cross section is even balanced out by the fact that the secondary and tertiary electrons produced during the ionization phenomena possess such high velocity that they also contribute to ionization. Hence the use of the same voltage and the same current source for collecting electrons and collecting ions, which simplifies the design considerably, is therefore important.

The rate of evacuation of the ionic pump section for argon can be easily estimated. The ionic current at the wall of the vessel is:

$$J_i = J_e \cdot P \cdot L \cdot Q_I \cdot \frac{n}{\pi}[A]$$

with the values:

Electron current $J_e = 0.3$ amp.
Oscillation factor $P = 10$
Length of the ionization chamber $L = 10$ cm.
Ionization cross section $Q_I = 4.5$ cm.$^{-1}$

and

Neutral gas pressure $p = 10^{-5}$ mm. Hg $\left(\frac{n}{\pi} = 10^{-5}\right)$

we get an ionic current at the wall of about $10^{-3}$ ampere. With a capture probability of 25% of the ions, the evacuation rate of the ionic pump section for argon is found to be 6 $L \cdot s^{-1}$ for the selected operating data at $10^{-5}$ mm. Hg. As the air contains about 1% of argon, the evacuation rate for getter-ion pumps of up to 600 $L \cdot s^{-1}$ evacuation velocity mentioned above suffices even if only argon is ionized.

The elements and the assembly of a high-vacuum system with getter-ion pump are shown in Fig. 3. It was found that the getter-pump action diminished critically or disappears completely of atmospheric air reaches the titanium metal-coating layers once or several times. That is why a titanium vapor collector sheet **13** is provided in the pump design shown in Fig. 1, which can be easily replaced and cleaned or renewed after atmospheric air has been in the pump for some period of time. This defect of the getter-ion pump, which was of a fundamental nature up to now, loses much of its importance if the invention's type of connection shown in Fig. 3, with a main vacuum valve **27**, a receiver **25**, and a forevacuum seal, is used. In this type of connection the preliminary evacuation of the receiver **25** is not done via the getter-ion pump, but directly. This makes it possible to close the main vacuum valve **27** at the end of an evacuation period before admitting air to the receiver, and to leave the getter-ion pump under high vacuum. Since the getter pump action persists for some time, as mentioned above, the pump itself sees to it that the gas load on the titanium collecting surface remains small, provided the main vacuum valve **27** is tight. This valve is opened to the next evacuation period only when the preliminary evacuation of the receiver **25** has reached a pressure lying between $10^{-3}$ and $10^{-4}$ mm. Hg. In Fig. 3 a smaller Root's pump **39**, combined with a two-stage rotating gas ballast forepump **34**, for example, is shown for this preliminary evacuation. As a rule, rotating pumps with comparatively low evacuation velocity will suffice. They are disconnected as soon as the prescribed forevacuum pressure has

been reached and the forevacuum valve 29 has been closed.

It was stated above that when the vaporizing pan is constantly heated the bottom opening in the baffle clogs up rapidly due to sintering of the falling powder because of the high radiation. This difficulty is eliminated in the invention by interrupting the heating of the vaporization cathode system during the brief period of titanium powder supply to the vaporizer. As soon as the quantity of titanium powder required for the individual vaporizing pulse has been supplied to the vaporizing pan, the vibrator circuit is opened, and at the same time the heating circuit, the vaporizer, and the cathode, are connected. After the end of the brief period of time required for vaporization, the current source at, for example, 800 volts, is connected to the grid 14. This adds the ion pump action to the vaporization getter action and to the initiated process of contact getter action.

In order to be able to evaporate as little getter metal as possible, it is advisable to make the frequency of vaporization pulses a function of the pressure in the receiver. It is therefore advisable to lower the receiver pressure to the desired value at the beginning of high-vacuum evacuation by means of a rapid sequence of vaporization pulses, after which a new vaporization pulse occurs only when the pressure rises again above a given value after the getter layer has been saturated. To make this operation automatic, a high-vacuum gauge 36 is connected to the receiver 25 as shown in Fig. 3, this gauge actuating a switch (pressure switch) as soon as the pressure exceeds a given value that can be set. This switch sets a program control mechanism 37 in motion, which controls the connection and disconnection of the various currents in the proper chronological sequence and for the proper length of time until the ionic pump process commences again.

26 is the high-vacuum gauge, 28 a forevacuum gauge, 30 a compensating valve, 31 a forevacuum gauge 2, and 32 a forevacuum valve, 33 is a drain pipe, 35 the getter-ion pump, and 38 the current source required. 40 is the water-vapor pressure, 41 the pressure of the other gases and vapors, and 42 the status of preliminary evacuation until the getter-ion pump starts. The occurrence of the getter pulses is represented by $n$ and $n+1$ and the ion pump operates in the interval between them.

Figure 4 shows the current source 43, the high-vacuum pressure switch 36 and the program controller 37 comprising motor 44, the speed regulator 45, the rotary switch 46 and relay 47 to provide a clear idea of the auxiliary equipment for the getter-ion pump.

The time sequence of operation and the successive operations involved are shown in Fig. 5.

The time sequence is:

(1) Measuring the pressure by means of the pressure switch 36.

(2) Feeding in the powder.

(3) Vaporization.

(4) Operation of the ion pump.

Thus Fig. 5 shows in graphs 1 to 5 the operating time of the pressure switch 36, rotary switch 46, the powder supply mechanism, the getter pulses applied to the cathode, and the ion pump. The uppermost curve shows the pressure in the receiver 25.

I have described a preferred embodiment of my invention, but it is understood that this disclosure is for the purpose of illustration, and that various changes in shape and proportion, as well as the substitution of equivalent elements for those herein shown and described may be made without departing from the spirit and scope of the invention as set forth in the appended claims.

What is claimed is:

1. An ion getter pump for producing a vacuum by means of the evaporation of a getter material and the ionization of the residual gas in an ionization space by electrons emitted by a cathode, comprising a housing, a grid electrode in the housing defining said ionization space, an integral evaporator cathode of refactory conducting material within the grid electrode having a cathode portion and an evaporator portion, means connected to the cathode for supplying heating current through the cathode, and means mounted in the housing above the evaporator portion of the cathode for supplying the getter material to the evaporator portion of said evaporator cathode; and electrical circuit means for controlling a chronological sequence of operations, said sequence controlling means comprising means connected to the getter material supplying means for feeding the getter material to the evaporator portion intermittently, means conneced to the cathode current supplying means for automatically interrupting the heating of the evaporator-cathode during the periods when getter material is being supplied to the evaporator-cathode, thus preventing partial sintering of the getter material, and means connected to the grid electrode for applying a voltage to the grid electrode after the vaporization of the getter material has been effected, for initiating an ion pump action superimposed on the vaporization getter action and the contact getter action.

2. An ion getter pump according to claim 1, wherein said voltage applying means is a source of voltage pulses.

3. An ion getter pump according to claim 2, wherein the ionization is produced by means including said cathode, said means for heating the cathode, a collector electrode adjacent the cathode and connected to said voltage applying means, and an electron mirror surrounding the collector electrode.

4. An ion getter pump according to claim 3, wherein the getter material is titanium powder and a titanium vapor collector electrode is replaceably mounted in the housing outside the grid electrode, whereby a diminution of getter pump action is avoidable by replacement of the collector electrode.

5. An ion getter pump according to claim 3 including additional means connected to said housing for producing a preliminary evacuation of the ionization space prior to the vaporization getter action and the ion pump action.

6. An ion getter pump according to claim 3, wherein the collector electrode is positioned so as to be heated by radiation from the evaporator-cathode.

7. An ion getter pump according to claim 1, wherein the getter material is a degassed metal powder.

8. An ion getter pump according to claim 7, wherein said metal powder is titanium.

9. An ion getter pump according to claim 8, wherein the evaporator-cathode includes a vaporization pan shaped and dimensioned so that the peripheral zone of the pan only reaches a temperature lower than the melting point of titanium while the central zone of the pan has a temperature higher than the melting point of titanium.

10. An ion getter pump according to claim 9, wherein said cathode is a tungsten cathode and said vaporization pan is positioned relative to said cathode so as to maintain as low a temperature gradient as possible between the cathode and the getter metal vaporizing surface of the pan.

11. An ion getter pump according to claim 10, wherein said vaporization pan is composed of graphite of maximum density and free of cracks.

12. An ion getter pump according to claim 11, wherein the vaporization pan is cup-shaped.

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