June 5, 1962 N. MILLERON **3,037,685**

METHOD FOR PUMPING GASES AT LOW VACUUM PRESSURES

Filed Oct. 16, 1959 3 Sheets-Sheet 1

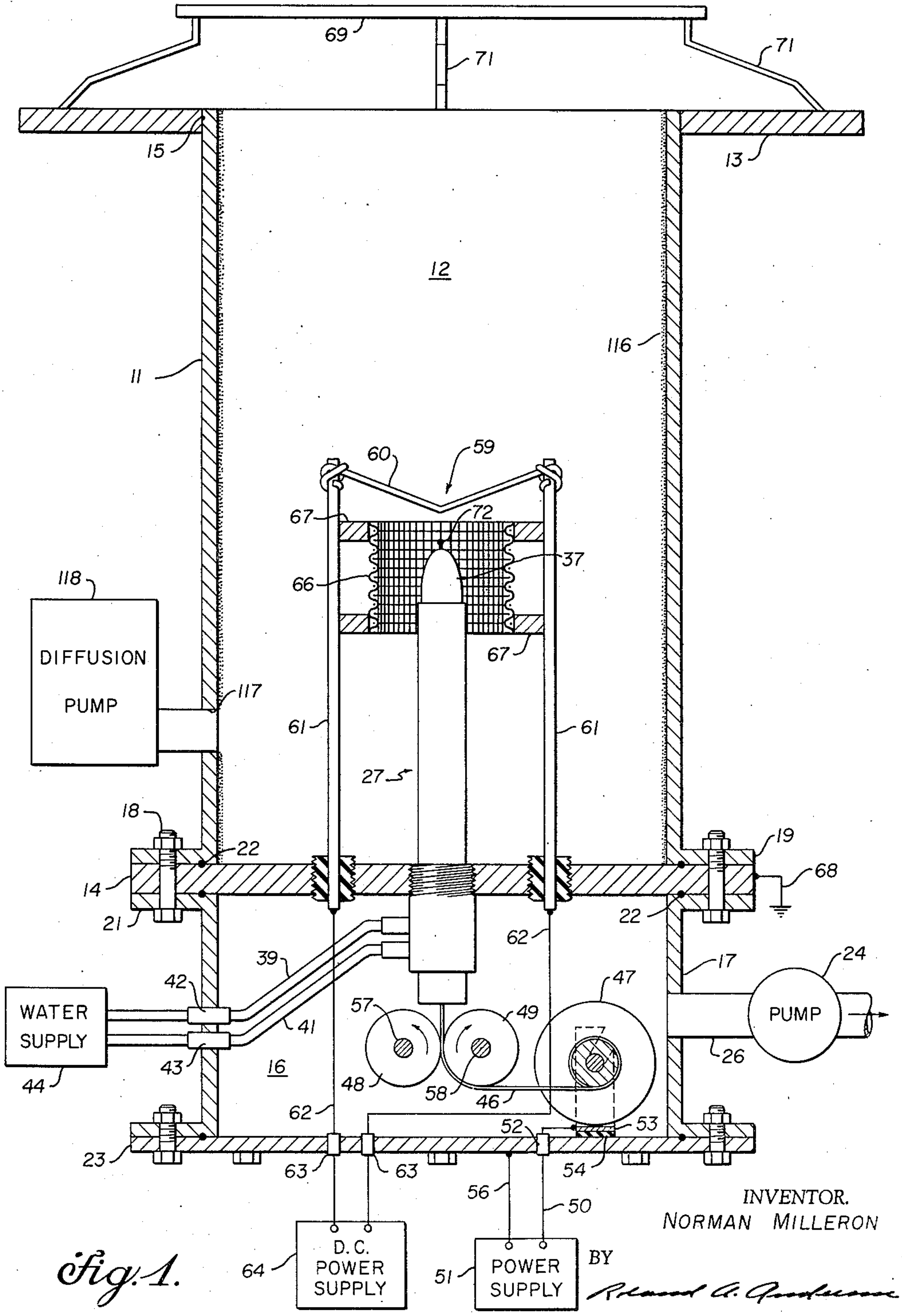

Fig. 1.

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Filed Oct. 16, 1959 3 Sheets-Sheet 2

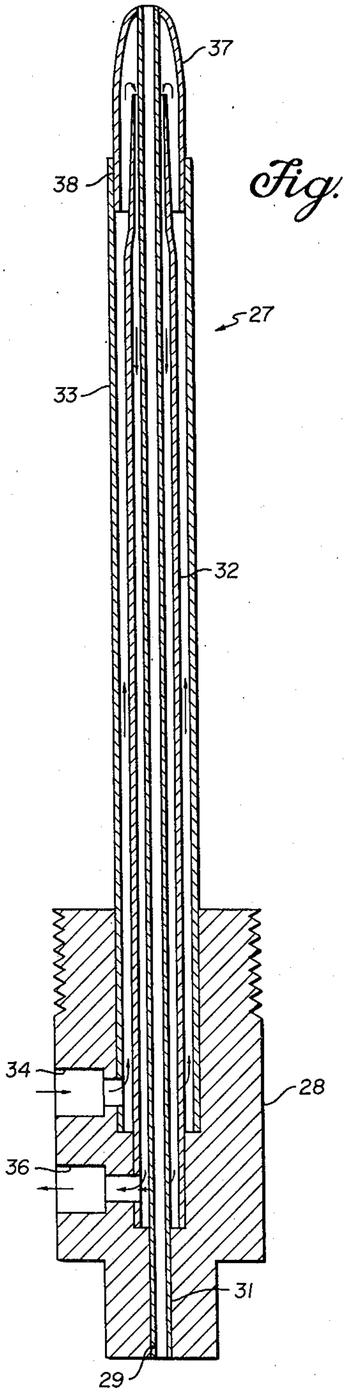

Fig. 2.

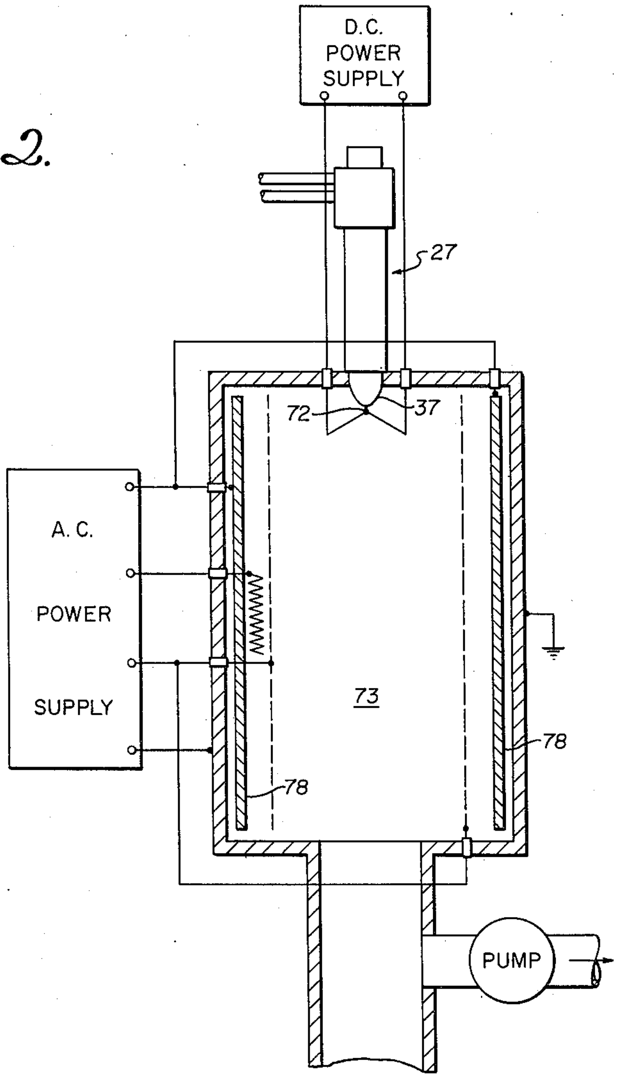

Fig. 3.

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Filed Oct. 16, 1959 3 Sheets-Sheet 3

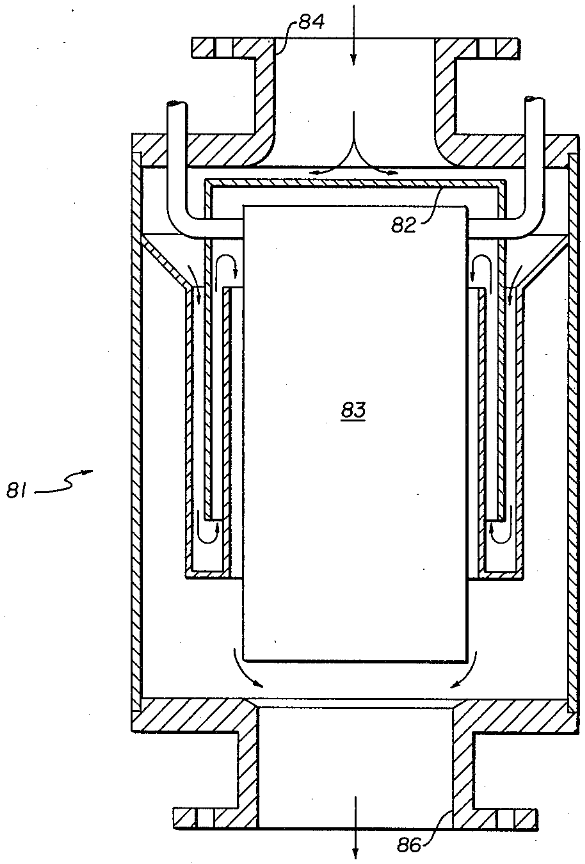

Fig. 4.

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3,037,685
Patented June 5, 1962

3,037,685

METHOD FOR PUMPING GASES AT LOW VACUUM PRESSURES

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Filed Oct. 16, 1959, Ser. No. 847,033
13 Claims. (Cl. 230—69)

This invention relates to an improved method of pumping gases at ultra low vacuum pressures. More specifically, this invention relates to a method of pumping overpressure "pulses" of gases having a vacuum pressure less than the base equilibrium pressure using a pump in which a part of the pumping cavity contains surfaces consisting of clean gettering metal.

By the term "gettering metal" is meant metals which ordinarily adsorb thereon large quantities of most gases commonly used in vacuum apparatus application. Unless indicated otherwise the term is meant to refer to such metal which has been at least partially degassed or baked out. Specific preferred gettering metals for use in the present invention are discussed hereinafter.

The invention provides a method of pumping overpressure "pulses" or "bursts" of gases without a significant rise in base pressure within a vacuum pump having surfaces within the pumping cavity coated with or comprising clean gettering metal, e.g., Mo or Ta. Means may also be provided for initially depositing additional clean metal or degassing said surface. The cavity is first pumped down by any convenient means to an equilibrium base pressure in the range desired, generally below 10^{-6} mm. Hg for most effectual use of the present invention. At this pressure, the metal immediately adsorbs overpressures or "bursts" of gases striking same with thermal motion without raising the base pressure significantly. Desorption takes place at an equilibrium rate which, of course, is dependent upon the equilibrium pressure, and such desorbed gases are continuously removed by diffusion pump or other pumping means, whereby said overpressures or "bursts" of gases are removed without a rise in the equilibrium pressure and/or back diffusion of the gaseous pulse from the pumping cavity.

In the low vacuum pressure art, vacuums lower than about 10^{-6} mm. Hg were generally not required or thought to be desirable prior to nuclear applications in the 1940's. Pressures of even this magnitude were found only in limited scientific research, generally where only low volumes were being pumped, i.e., hundreds of liters per second or less. For this purpose mechanical pumps and diffusion pumps were admirably suited. However, in the present technology of mass spectroscopy, particle accelerators, controlled thermonuclear reactions, electronic applications, space research, and associated fields, threshold pressures are frequently much lower. For example, random gas particles may produce erroneous results in mass spectroanalysis of extremely small or pure samples, and nuclear reaction thresholds may be contingent upon pressures as low as 10^{-10} mm. Hg or lower.

Typical optimum requirements for vacuum pumps in modern technology are exemplified in plasma containment experiments, accelerator systems and ion sources in which an almost absolute gaseous sink relationship must be maintained. In such embodiments, energetic charged particles and energetic neutral particles are continually introduced or produced in the system. The vacuum pump or "sink" must be capable of both initial evacuation and consequent removal of the total output of desirable fast particles and an irreducible amount of slow neutral particles to maintain a very low density of "cold" neutral gas. Optimumly, the fast particles should be removed without initial conversion to slow ones, an operation not possible with diffusion pumps, since fast particles are difficult to entrain by the jets of a diffusion pump. Further, when fast particles are first reduced to slow ones, a larger entrance orifice must be provided, since fast particles can be "shot" through a small aperture, while slow ones can not.

One method and apparatus by which vacuums as low as 10^{-10} mm. Hg and lower have ben established and maintained while pumping the equivalent of thousands of liters per second has been the use of gettering or ion gettering pumps in which the pumping cavity and/or collecting electrodes are continuously coated with degassed or clean gettering metal vaporized from a source disposed within the pumping cavity adjacent the collecting surfaces. Gases entering such cavities and striking the walls or collecting plates thereof are adsorbed and "buried" by the fresh layer of desorbed gettering metal continuously deposed thereon.

One such pumping embodiment is disclosed in United States Patent No. 3,024,965, entitled Apparatus for Vacuum Deposition of Metals by Norman Milleron, filed October 8, 1957, wherein a wire or strip of suitable gettering metal is continuously fed into a pumping cavity through an externally cooled feeder tube adapted to thermally shield the wire or strip and constructed of a material having a high enough coefficient of thermal conductivity that heat contained or generated in the tube surface is removed. Upon emerging from the top of the cooled tube into the pumping cavity the wire is subjected to an electron beam bombardment or other heating means causing the wire outside the feeder tip to be heated and melted. The emerging wire is preferably disposed in a vertical position to ensure symmetrical distribution of metal by gravitational force and it is further disposed generally perpendicularly to the plane of the electron gun or heat source in order to facilitate melting primarily in the outermost tip of the wire. The molten metal tends to form a molten ball held together by surface tension forces (i.e., surface tension), when the coolant, wire feed and heating means are properly regulated. As used herein the term "molten ball" is meant to include any mass of molten metal on the tip of a wire which is being acted upon by surface tension. Heat contained in the wire behind the molten ball is conducted to the feeder tip or tube primarily by radiation rather than conduction. The molten metal in the ball is radiated outwardly in a "line of sight" direction, while at the same time additional metal from the feeder wire is melted to replenish the molten mass. The gettering metal wire is, of course, degassed prior to introduction into the vacuum cavity, and close tolerances are maintained between the feeder wire and the feeder tube.

As supplied to a simple gettering pump, gaseous adsorption on the continuously deposited metal coating is employed to create a very high vacuum, usually after the vacuum cavity has initially been evacuated to a pressure of about 10^{-4} mm. Hg with a fore pump, i.e., the particles remaining in the cavity impinge randomly upon the walls of the pumping cavity connected with the container to be evacuated and are held thereon by the continuously increasing coat of adsorbent metal.

In the application of the gettering concept to ion pumps, gaseous particles to be evacuated are permitted to randomly enter an area between two cathode plates. A stream of electrons passing between the cathode plates, or other means, ionizes the gaseous molecules and the resulting ions are attracted to the cathode plates. Ordinarily the ions are free to leave the plates upon contact therewith since they lose their charge and become neutral molecules. However, by placing the vaporized metal source described hereinabove within the "line of sight" of the contact surfaces of the cathode plates, the vaporized metal is coated thereon and the ions attracted thereto upon neutralization are adsorbed or "buried" within the continuously deposited metal.

By employing the above-described embodiments, in combination with gettering metals such as titanium, tantalum, tungsten, molybdenum, zirconium, niobium, rhenium, thorium and uranium, which have negligible vapor pressures, vacuum pressures as low as or lower than 10^{-10} mm. Hg can be attained while pumping at rates equivalent to hundreds of thousands of liters per second. However, in many pumping situations, such as those described hereinabove, very low vacuums must be maintained over extended periods of time. As practiced in the past very extensive coats would be built up upon the cavity surfaces during this period, even through the actual number of gas particles actually captured might be relatively small.

It has now been discovered that, because of the low pressure adsorption capacity and adsorption-desorption equilibrium rates of certain gettering metals at low vacuum pressures, equilibrium pumping pressures lower than 10^{-6} mm. Hg may be maintained, and short overpressure pulses may be removed, without the continuous disposition of fresh gettering metal onto the pumping surfaces by the method of the invention. Specifically, a vacuum pumping cavity having a gettering metal pumping surface is initially either coated with fresh metal or degassed, and the cavity and/or adjacent vocuum chamber is evacuated to a pressure of the order of 10^{-6} mm. Hg or lower. At this point, if not previously, a diffusion pump equipped with a cold trap to preclude back diffusion of oil vapors and capable of pumping moderate volumes at pressures as low as 10^{-8} to 10^{-10} mm. Hg is connected in series with the main pumping cavity and permitted to pump continuously upon the gettering metal surface and pumping cavity, whereby a base equilibrium pumping pressure is established, depending upon the diffusion pump base pressure, and upon leaks into the pumping system and upon other factors. Equivalent pumping means may also be used in place of the diffusion pump. Fresh gases entering the pumping cavity strike the gettering metal surface and are immediately adsorbed; desorption rates are in the range of 10–100 particles/cm.^{2}/sec. for relatively clean or unsaturated metals at pressures in the 10^{-8} to 10^{-10} mm. Hg range, permitting evacuation of the gases degassed at this rate with the diffusion pump. Therefore, when overpressures or "bursts" of gases or ions enter the cavity for brief periods, as from a pulse of accelerated particles, they strike the gettering metal surface and are adsorbed completely, without a significant rise in the equilibrium pumping pressure as long as the adsorption capacity of the metal is not exceeded. When the overpressure ends, the gases continue to degas at the same rate, or at only a slightly higher rate, permitting complete evacuation of the gas overpressure without significant rise in the equilibrium pressure.

The invention permits vacuum pumping at pressures below 10^{-6} mm. Hg by desorption of gettering metal surfaces with a diffusion pump while most entrant gases are adsorbed thereon at the same pressure. This is accomplished without special heating of the gettering surfaces, as usually done in order to degas vacuum equipment. When the pressure of the newly admitted gas rises above the pressure at which the metal was previously degassing, the gettering metal continues to adsorb gases and because of its large capacity for these gases it does so almost indefinitely. The desorption-adsorption equilibrium shifts only very slowly so that after the additional gas from an overpressure pulse has been adsorbed, the desorption pressure remains almost at the pressure at which it had been desorbing prior to the receipt of the pulse of gas. For example, it has been found that the walls of a closed box of 7500 cm.^{2} surface area at a pressure as high as 1×10^{-7} mm. Hg can pump as much as 0.1–0.2 micron liter of hydrogen and yet desorb this gas completely at a net rate determined by the conductance of the diffusion pump system.

Accordingly, an object of the invention is to provide a method for pumping gases at low vacuum pressures using gettering, ion gettering or similar equipment.

Another object of the invention is to provide a method of pumping overpressures or "bursts" of either slow residual particles or accelerated particles without a substantial rise in the equilibrium or base pressure.

A further object of the invention is to provide a method for pumping overpressures or "bursts" of particles by adsorption onto a gettering metal in equilibrium with a base pressure below 10^{-6} mm. Hg, whereby said equilibrium pressure is raised only slightly because of the capacity of the metal for gases, and thereafter removing said gases with a diffusion pump as they are desorbed from the gettering metal.

Another object of the invention is to provide a method for reversably adsorbing and desorbing gases from clean gettering metal surfaces at a low base pressure to provide a constant pumping action.

Another object of the invention is to provide a method for continuously pumping gases with a gettering or ion gettering pump having means for continuously coating a gettering surface with fresh gettering metal without such continuous deposition, even at pressures below 10^{-6} mm. Hg and even though overpressures of gas or "pulses" of accelerated particles enter the pumping cavity.

Another object of the invention is to provide a method for pumping gases in a gettering pump, ion gettering or other type pump in which a collecting surface is coated with a layer of clean gettering metal by pumping upon said collecting surface with a diffusion pump capable of pumping to pressures in the range of 10^{-8} mm. Hg or lower, whereby the metal is gradually desorbed even though the metal reversably adsorbs gases at the same pressure. By pumping continuously with the diffusion pump, a uniform low pressure is retained within the vacuum cavity, even though large pulses of gases are intermittently adsorbed into the metal at a rate faster than the desorption rate.

Additional objects and advantages of the present invention will become apparent from the ensuing description taken in conjunction with the accompanying drawings, of which:

FIGURE 1 is a cross sectional elevation view, partly in schematic, of a preferred embodiment of an apparatus for the vaporization and continuous vacuum disposition of metals and particularly adapted for use as a gettering pump in which the method of the invention may be practiced;

FIGURE 2 is a longitudinal cross section of the fluid cooled unit of FIG. 1;

FIGURE 3 is a partially schematic cross sectional view illustrating an ion pump embodiment in which provision is made for the continuous vaporization and plating of metals upon the cathode plates and in which the method of the invention may be practiced; and

FIGURE 4 is a cross sectional side view of a liquid nitrogen cold trap adaptable to connection to the inlet of an oil diffusion pump.

In the practice of the invention there will ordinarily be provided a cavity or container capable of low vaccum pressure containment and adaptable for connection to the pump employed in the pumping method of the invention. Initially the container may contain air or some other gas at amtospheric pressure, but where the invention is to be used to greatest advantage, i.e., maintenance of vacuum pressures in large containers in the vacuum region below 10^{-6} mm. Hg, the container will most frequently be pre-evacuated under conditions bringing about degassing of wall surfaces and other surfaces within the container, as with a diffusion pump. Where the vacuum cavity or container is being used for experimentation or activity in many types of endeavor, such as vacuum space technology, outer space studies, mass spectroscopy, plasma containment, nuclear studies and others, particles other than air or ordinary gases will also tend to be present together with residual or degassed impurities. Such particles and gases frequently include gaseous ions, elementary energetic particles and lightweight gaseous materials such as diatomic or dissociated hydrogen, deuterium, and tritium. These particles and gases may have random thermal motion, but frequently will have an initial directional velocity, over a spectrum of energies rendering immediate vacuum capture difficult or uncertain. Vacuum capture and ejectment with negligible back diffusion into the vacuum cavity proper is especially difficult in practice where the aforementioned light gaseous particles, hydrogen, deuterium and/or tritium are present, since the random thermal motion velocities even in relatively unexcited states are extremely rapid. Obviously in order to attain the most favorable equilibrium, the number of "bounces" or collisions between gas particles or with non-capturing surfaces must be held at a minimum since the probability of back diffusion thereafter is quite large.

The critical conditions under which vacuums must frequently be maintained are exemplified in plasma containment devices wherein charged particles are contained within a suitable arrangement of electric and magnetic fields housed within a vacuum chamber. Generally, the vacuum container must be evacuated to a base pressure of 10^{-9} mm. Hg or lower, exclusive of the centrally contained plasma. Since charged particles may not be inserted directly into the central plasma region because of the restraining magnetic field, various methods of injecting neutral particles are used. For example, directional beams of energetic neutral particles such as hydrogen, deuterium, and tritium may be obtained through collision of energetic beams of charged particles with neutral particles or by charge exchange with charged particles, whereby entrance into the containment region may be made through the electric and magnetic fields thereof. Ions and other particles are created within the containment region by plasma charge exchanges or by nuclear reactions, ionizing collisions, etc. Some of the neutral particles pass completely through the containment region and must be evacuated together with charged particles, degassed impurities, etc.

For most efficient operation in plasma work of the type described, the pumping means is disposed in parallel alignment to intercept the residual beam of charged and/or neutral particles, so that most of the particles or gases are trapped prior to the time when they slow down and wander randomly throughout the container. The vacuum cavity may be integral with the pumping cavity, but where pumping against a beam of particles, as aforementioned, a wide mouthed aperture of some depth is preferred to facilitate containment prior to the initial collision. Such problems are not peculiar to plasma containment alone, but are encountered in accelerator and other high energy particle fields. Where random or thermal motion is relied upon, as in fields of endeavor where charged particles are normally absent, to introduce gases into the pumping cavity, an aperture wide in relation to diameter or width of the pumping chamber is also preferred in order to increase the probability of entrance.

In accordance with the process of the invention, stated in simplest terms and shown generally in FIG. 1, there is provided a vacuum housing **11** communicating through opening **15** in vacuum tank **13** to which it is joined in vacuum tight relation. The pump housing **11** defines a pumping cavity **12**, and the inside surface **116** of housing **11** comprises a gettering metal, which may be provided by vapor deposition means **27**. Adjacent to the pumping surfaces **116** and generally at the opposite end of the pumping cavity **12** from entrance **15** is outlet **117** leading to pump means such as diffusion pump **118**.

In the practice of the invention the gettering metal

pumping surface **116** is generally first degassed and the pumping cavity is pumped down by the pumping means such as diffusion pump **118**, usually together with the vacuum cavity to be evacuated, to a pressure below 10^{-6} mm. Hg. Most frequently the pumping surface **116** will be further continuously coated with gettering metal using continuous vapor deposition means **27** to obtain a pumping action by which the vacuum cavity is pumped down to a base or equilibrium pressure. Alternatively, by prolonged pumping, the equilibrium pressure may be attained with diffusion pump **118** alone. At the end of this period, which may include degassing by heating means, the base pressure is maintained with the diffusion pump **118** alone. Ordinarily, the diffusion pump will be conventional in design except for a cold trap or other trap means to prevent back diffusion of oil or pump fluid at lower pressures.

After the base pressure desired has been attained, the operations within the vacuum cavity are initiated, e.g., the pumping cavity is opened to the cavity to be evacuated, or if already opened the experiments or applications within the vacuum cavity are commenced. The pumping cavity is thereby exposed to overpressures or bursts or pulses of particles produced in the vacuum operations. The diffusion pump is maintained in continued operation, but no additional gettering metal is applied to the surfaces. As random amounts of particles enter the pumping cavity and strike the gettering metal surfaces by thermal motion, they are adsorbed and desorbed according to the equilibrium thereof, and eventually by random motion enter the diffusion pump inlet and are removed. "Bursts" of particles or overpressures pulses of particles of short duration entering the pumping cavity also strike the gettering metal surfaces. Instead of a pressure rise commensurate with the magnitude of the overpressure, however, the entire quantity of particles are adsorbed almost immediately—after only a few bounces against a gettering metal surface per particle—until the surface becomes saturated. The desorption rate, instead of rising commensurate with the increased number of particles adsorbed, increases only non-linearly by a few percentile of the overpressure because of the remarkable newly discovered affinity of gettering metals for large amounts of gases at low pressures. By continued pumping with the diffusion or other pumping means on the gettering metal surfaces, then, the additional gases adsorbed because of the overpressure are removed as they are desorbed without a significant increase in the base equilibrium pressure.

The ability or affinity of the gettering metal to adsorb the overpressure is limited only by the capacity of the metal for gases, which capacity is about 10^{14}–10^{15} particles per square centimeter of collecting surface. The ability of a given embodiment to adsorb all of the particles of an overpressure will then depend upon the surface area of the gettering metal available. In practice overpressures larger than the base pressure by a factor of 10 or 100 or more are completely adsorbed and thereafter removed at a rate dependent upon the conductance of the diffusion pump.

Specifically, using the metals Mo, W, Ta, Zr, Ti, Nb and others as gettering metals on a surface in the form of a coating at least 10^{-5} cm. thick, an amount of gaseous hydrogen equivalent to $\sim 10^{17}$ particles can be adsorbed onto each square centimeter of wall surface at a base pressure of about 10^{-8} mm. Hg without raising the base pressure by the adsorption-desorption equilibrium to more than about 10^{-7} mm. Hg. The entire amount of gas is readily adsorbed after only a few "bounces" per molecule against the gettering metal surface. The adsorption-desorption equilibrium for gases other than diatomic hydrogen is altered even less under the above circumstances since they are easier to adsorb and less readily desorbed. Other gettering metals which might be used but for which precise data does not exist are Ba, Al, and Rh.

Referring now to FIG. 1 there is shown a preferred embodiment of an apparatus used in the practice of the invention in which a pumping cavity is adapted to the remote disposition of gettering metal upon the walls thereof and also for continuous evacuation with a diffusion or other pumping means. This invention is described in the aforementioned U.S. Patent No. 3,024,965, by Norman Milleron, filed October 8, 1957 entitled Apparatus for Vacuum Deposition of Metals, in which like elements have like numbers in the drawings. Specifically, there is shown an elongated cylinder **11** defining a vacuum chamber **12** opening at the upper end into a vacuum tank (not shown) formed by walls **13**. The bottom of chamber **12** is closed by removable base plate **14** which also serves as an end wall to a second vacuum chamber **16** defined by cylinder **17**. A pressure fit is provided between cylinders **11**, **17** as by means of bolts **18** acting upon flanges **19**, **21** of such cylinders **11**, **17** respectively, upon either side of base plate **14** and upon deformable metal gaskets **22** interposed therebetween. The second chamber **16** is suitably closed at the lower end by a removable backing plate **23**, and is preferably connected with a fore pump **24** through exit pipe **26**.

A vertically disposed water cooled feed unit **27** is coaxially mounted within chamber **12** as by a threadable engagement with base plate **14**. The feed unit **27** is shown in cross section in FIG. 2 and consists of a threaded massive plug member **28** adapted to engage base plate **14** and through which member extends a bore **29** generally perpendicular to the plane of the exterior threads. A central elongated metal feed tube **31** in close fitting relation to bore **29** extends the length thereof and for a distance into vacuum chamber **12**. Suitable tubes **32** and **33** are respectively disposed outwardly concentric with respect to feed tube **31** and both are countersunk for a distance into the top of plug member **28**, outermost tube **33** being countersunk to a leser depth than intermediate tube **32**. Inlet and outlet ports **34** and **36** below base plate **14** in plug member **28** provide coolant entrance and exit means into tubes **33** and **32**, respectively. A suitably shaped tip **37** is joined to outer tube **33** as shown generally at **38** by any appropriate means of rigid attachment and extends the tube structure to form a union with feed wire tube **31**. Coolant water or equivalent cooling fluid introduced into inlet port **34** flows upward between tubes **32** and **33**, is deflected by tip **37** and flows downward between tubes **32** and **31** and out exit port **36**. Pipes **39** and **41** are preferably connected to inlet and exit ports **34** and **36**, respectively, and lead exteriorly through chamber wall **17** as shown generally at **42** and **43**, respectively, to facilitate connection to an external water supply **44**.

A feed wire **46**, fabricated from any desired gettering metal to be deposited, is wrapped on a rotatable metal spool **47** and passes between conventional exteriorly powered feeder rollers **48** and **49** to facilitate continuous introduction of the feed wire into feed wire tube **31**.

While numerous conventional means may be used to maintain the vacuum around rotatable drive shafts **57** and **58** extending through wall **17** for connection to rollers **48** and **49**, respectively, the use of liquid metal seals has been found particularly convenient and leak proof, for example, a metal having composition of 62.5% gallium, 21.5% indium and 16% tin, which melts at approximately 10° C., has been utilized to great advantage in practice.

Reasonable vacuum integrity is maintained between vacuum chambers **12** and **16** communicably connected through feed wire tube **31** by selection of a feed wire size having a tolerance of 5 mils or less with respect to the feed wire tube **31**. In addition, degassing of the wire **46** prior to feeding into the water cooled feed unit **27** is accomplished as by resistance heating which is preferably produced by means of a conductor **50** connected to a power supply **51** and led through backing plate **23** at a vacuum insulated point **52** to be connected to a spool holder **53** for supporting spool **47**. Holder **53** is made of a conducting metal and is insulated from the backing plate as shown generally at **54**. Power supply **51** is grounded to the backing plate by contact wire **56** to complete a circuit to ground through a current conduction path including wire **46**. The wire is thus resistance heated by virtue of the current flowing therethrough. It will be appreciated as regards the fabrication of material of feed wire **46** that such wire may be gettering metal or combination of same. For adsorption and pumping processes during continuous vaporization of the feed wire and deposition thereof in the pumping cavity thereafter titanium, tantalum, tungsten and molybdenum, also zirconium, niobium, thorium and uranium, have been found superior.

In order to heat the feed wire **46** emerging from the top end of feeder tube **31** to a high temperature, means are provided for bombarding the emerging feed wire with an electron beam. The foregoing means may be provided, for example, as an electron gun **59** having a heated cathode filament **60** mounted above tip **37** as by means of cathode posts **61** embedded in and insulated from base plate **14**. Energization of filament **59** to supply electrons is facilitated by conductors **62** respectively connected to posts **61** at the under side of base plate **14**. Such conductors are led exteriorly through hermetically sealed bushings **63** mounted in backing plate **23** for connection to a suitable power supply **64** operating above ground potential. The electrons emitted by filament **60** are roughly focused onto the molten ball or mass **72** by a bias screen **66** supported coaxially about tip **37** by electrically conducting supports **67** attached to posts **61**. Screen **66** is thereby maintained at least as negative as the potential of power supply **64** and therefore filament **60**. Moreover, feed wire **46** is grounded through base plate **14** as shown generally at **68** whereby the potential gradient thus established between filament **60** and the emerging tip of feed wire **46** is effective in accelerating the beam of electrons thereto. The impinging electron beam vaporizes the tip of the feed wire **46** as hereinafter described with respect to the operation of the invention. To prevent the metal vapors thus evolved from contaminating vacuum areas outside of the main pumping cavity **12** a circular plate **69** is best disposed in spaced relation to wall **13** by means of supports **71** anchored thereto. All of the foregoing embodiment as exemplified by the drawings is described in the aforementioned copending application and serves as preferred structure in the process of the invention.

In the process of coating the container walls or other surfaces within the pumping cavity **11** preparatory to the method of the invention and optionally at the same time of evacuating the chamber in the event the diffusion pump is not used, as discussed hereinbefore, the vacuum deposition apparatus is operated as follows: The vaporization chamber **12** is first pumped down by conventional means such as a fore pump (not shown) to the pressure of 10^{-4} mm. Hg or less to avoid direct discharge when the electron gun is energized. The cooling water is next circulated through feed unit **27**, water of room temperature or even higher being sufficient as long as the flow within the chamber **12** is sufficient to remove heat rapidly. Feeder rollers **48** and **49** are activated and filament **60** is energized. Once the filament **60** has been energized the stream of electrons arriving at the emerging feed wire **46** heat the tip of the wire and cause it to become molten. A self-supported molten mass or ball **72** forms at the tip of the wire, which ball is held together by surface tension. The molten ball **72** shields the feeder tip **37** from electron bombardment. Sufficient energy is "rained" (concentrated) on the surface of the molten ball **72** to cause it to evaporate at a rate that is matched by the rate of feed of the wire. The feed wire immediately behind the ball is thermally and electrically protected by the water cooled tip **37** without which the ball **72** has a strong tendency to burn off because the wire becomes thin and is unable to support the molten ball.

When the vaporization apparatus described hereinabove is operated merely as a means of vapor coating the pumping cavity surface **11** with a gettering metal, as one initial step in the present pumping process, the feed wire mechanism and electron beam gun are merely operated in cooperation until such time as a coating of the desired thickness is built up, at which time the respective mechanisms are de-energized. A coating at least 10^{-5} cm. thick is generally sufficient for the purposes of the present invention. Most frequently the coating will be applied on top of an older coating applied in a previous pumping operation, since it is quicker and easier to apply fresh clean coats with the preferred embodiment than to degass the older coatings to low vacuum pressures. In most instances the entire system will be pumped down to a pressure with the apparatus using the advantaneous gettering action of the freshly deposited metal. This method of pumping down the vacuum cavity is preferred for the reason that it is much quicker than other methods and for the reason that this is a function normally supplied by the preferred gettering apparatus. At the end of this pumping period sufficient fresh coating will have in all instances been applied so that further pumping in the low pressure region after the gettering pump proper is shut down is obtained by the method of the invention described in detail hereinafter.

Operation of the meal coating or gettering apparatus described hereinabove as a preliminary step to pumping by the method of the invention is accomplished by first pumping out the upper and lower vacuum chambers **12** and **16** for extensive periods of time at pressures of 10^{-4} mm. Hg or less to degass the gases normally entrapped on the walls thereof. During this preliminary pumping period, or during later operation, power supply **51** provides means for heating the feed wire **46** to a temperature of the order of 2000° C. by resistance heating, the exact temperature depending upon the melting point of the particular wire material being used. All gaseous impurities are normally degassed at this temperature and the feed wire **46** will then have a purity commensurate with that of the original metal. The entire vaporization unit is next brought into operation as detailed previously and vaporized metal continuously travels in a line of sight from the molten ball to the walls and other surfaces disposed within the chamber **12**. The clean pure metal coats the walls continuously and adsorbs and covers gaseous molecules thereon by chemisorption and physical adsorbtion mechanisms.

In accordance with the present invention, other methods may obviously be used to pre-coat the vacuum pumping cavity walls or as stated previously walls composed of a gettering material may be initially provided and thereafter cleaned of adsorbed gaseous materials by degassing at an elevated temperature. Additionally, the above-described wire feed and metal vaporization unit **27** may be advantageously applied to other types of vacuum pumps than the simple gettering pump described. Specifically, the pumping capacity of a conventional ion pump, for example of the Penning Discharge type shown in FIG. 3, with flat cathode collector plate electrodes is materially increased by the incorporation therein of a water cooled feed unit **27** as previously described. Such feed unit **27** is best disposed with the water cooled tip **37** thereof in a vertical position within the ion pump cavity **73**. The molten ball **72**, when formed by the feed unit, is preferably in a line-of-sight distance from all parts of the opposing surfaces of the electrodes **78**. The electron source and all other accessory means of the metal vaporizing source may be the same as that shown in FIG. 1. Operation of such pumps is described in detail in the aforementioned copending United States Patent Application.

The foregoing embodiments have been described as

preferred embodiments of apparatus for providing a pumping cavity having a surface therein composed of a gettering metal, for use in the process of the invention. In addition, a secondary pumping means in series with cavity **12** though outlet **117**, capable of pumping to the base pressures desired, must also be provided. Since this source should be capable of pumping large quantities at the stated base pressure, a conventional oil diffusion pump **118** is generally the most convenient means. In applications where the pumping method of the invention is to be used to greatest advantage, i.e., pressures below 10^{-6} mm. Hg, the diffusion pump must be supplied with means to prevent back diffusion of oil molecules into the pumping cavity. Several cold traps serving this purpose are known in the art and are capable of preventing back diffusion to pressures of the range of 10^{-9} mm. Hg. Specifically one such barrier trap **81** is shown in cross section in FIG. 4, the arrows indicating the line of gas flow around a conduction cooled barrier **82** and against a liquid nitrogen pot **83**, whereby gas laden with oil entering at inlet **84** traverses the maize in which the oil condenses out and finally the gas emerges at outlet **86**.

In the practice of the process of the invention using the preferred embodiments described hereinabove, the pumping cavity and the main vacuum chamber are first evacuated to the base pressure desired using the gettering pump or the ion pump embodiment having provision for continuous disposition of gettering metal onto the electrode collector plates. During this period the operation of the diffusion pump is not necessary. Once the base pressure is attained, the gettering and/or ion pumping means is turned off and the diffusion pump is relied upon. For most advantageous operation of the method of the invention, the base pressure will at this time be in the range of 10^{-7} to 10^{-9} mm. Hg or lower. At this pressure range the degassed gettering metal surface readily absorbs large quantities of gases when an overpressure occurs, i.e., a pressure increase by a factor of ten or more, because of the unexpected affinity which has been discovered of the gettering metal under pressures at which only desorption ordinarily would be expected to occur, as discussed hereinbefore. Upon adsorption of the "burst" or overpressure pulse from the main vacuum chamber the gettering metal immediately begins to desorb again. By appropriate selection and/or regulation of the conductance of the oil diffusion pump in relation to the pumping cavity volume, the diffusion pump removes substantially all of the desorbed gases from the gettering metal and continuous operation of the gettering pump over long periods of time is not necessary to insure an ultra-low vacuum. Use of the gettering metal surface aids the diffusion pump because there is less opportunity for "bursts" to back diffuse into the main vacuum chamber. The gettering pump may be turned on for brief periods if necessary to accommodate exceptional pumping requirements, as where a leak to the external atmosphere develops, or where additional containers are cut into the system. In such instances the base pressure will surge upward by factors of 10^3 or more and the physical volume of new gases will be larger than the gettering metal can accommodate.

Example

A 70 liter volume stainless steel vacuum tank was baked out at 400° C. and evacuated by a 4 in. oil diffusion pump. A base pressure of 10^{-9} was obtained using the diffusion pump alone when the intake was cooled by a liquid nitrogen cooled trap of slightly different structure but having a function similar to the one described hereinabove. After evaporation of molybdenum from a gettering pump identical with the embodiment hereinabove described within the tank for about one hour, the system pressure dropped to abtout 1×10^{-10} mm. Hg. In testing the pumping capacity of the system, pulses of hydrogen gas were introduced into the tank without operation of the gettering pump and the following data were obtained:

Volume defined by tank	~70 liters.
Projected area of coated wall	~7500 cm.^{2}.
Specific area measured by low temperature adsorption on Mo coating	>20 times projected area.
Base pressure	$<10^{-10}$ mm. Hg.
Amount H_2 delivered/pulse	$>4\times10^{16}$ particles.
Time duration of pulse	<1 second.
Max. pressure rise/gas pulse (walls newly active)	1×10^{-8} mm. Hg.
Indicated equivalent pumping speed	4×10^4 liters/sec.
Amount of H_2 to saturate coating, i.e., rise pressure to 1×10^{-5}	~1 mm.-liter.

After the above data were checked out, the gettering pump unit was turned off and hydrogen gas bled into the system until the gas pressure was about 1×10^{-5} mm. Hg, at which times no more hydrogen gas was permitted to enter. Pumping was then maintained with the diffusion pump alone. The diffusion pump was observed to take 5 seconds to reduce the system pressure from 2 to 1×10^{-6}, 53 seconds from 2 to 1×10^{-7}, and 570 seconds from 2 to 1×10^{-8} mm. Hg. Pulses of hydrogen of magnitude previously specified, i.e., 4×10^{16} particles, were then introduced into the tank. In each instance the pressure rise was only nominal, of the order of 1×10^{-8} mm. Hg after introduction of the initial pulse, indicating that hydrogen was completely adsorbed almost instantaneously by the gettering surfaces. Desorption and removal was againt brought about by pumping with the diffusion pump for the indicated periods, the rate being determined by the conductance of the diffusion pump system.

While the invention has been disclosed with respect to several preferred embodiments, it will be apparent to those skilled in the art that numerous variations and modifications may be made within the skill of the art and thus it is not intended to limit the invention except as defined in the following claims.

What is claimed is:

1. In a method for pumping and removing gas particles at low vacuum pressures comprising the steps of establishing a vacuum pressure equilibrium at a pressure lower than about 10^{-6} mm. Hg within a cavity to be evacuated, said cavity having a gettering metal surface disposed contiguous with said cavity space in adsorption-desorption equilibrium therewith, subjecting said cavity to a gaseous overpressure, whereby at least a portion of said particles are adsorbed, and thereafter removing gases desorbed from said gettering metal surface and gases above said equilibrium pressure from said cavity with conventional pumping means having a pump inlet adjacent said gettering surface.

2. In a method for pumping and removing pulses of gases from a cavity at low vacuum pressures comprising the steps of establishing a vacuum pressure equilibrium at a pressure below about 10^{-6} mm. Hg within a pumping cavity adapted to communicate with a chamber to be evacuated, said pumping cavity having a gettering metal surface disposed contiguous with said cavity space in adsorption-desorption relation with said cavity, subjecting said cavity to a gaseous overpressure, whereby gases are adsorbed from said gaseous overpressure onto said gettering metal surface, and thereafter removing gases desorbed from said gettering metal surface and gases above said equilibrium pressure from said cavity with a diffusion pump having a pump inlet adjacent said gettering surface.

3. In a method for pumping and removing overpressures of gases at low vacuum pressures from a vacuum cavity comprising the steps of evacuating and establishing a

vacuum pressure equilibrium at a pressure below about 10^{-6} mm. Hg within said cavity to be evacuated, remotely renewing a gettering metal surface within said cavity with a coating of desorbed gettering metal, adsorbing gases from said gaseous overpressures onto said gettering metal surface whereby said overpressure is reduced substantially to said equilibrium pressure, and thereafter removing gases desorbed from said gettering metal surface between overpressures with conventional pumping means having a pump inlet adjacent said gettering metal surface.

4. In a method for pumping and removing overpressure pulses of gases and gaseous particles from a vacuum cavity comprising the steps of evacuating and establishing a vacuum pressure equilibrium at a pressure below about 10^{-6} mm. Hg within a pumping cavity in which overpressure pulses of gases are periodically released in volumes greater than a conventional diffusion pump connected thereto can immediately remove, remotely renewing a gettering metal surface disposed within said pumping cavity to receive thereon gaseous molecules from said overpressure pulses, adsorbing gases from said overpressure pulses onto said gettering metal surface whereby said overpressure is reduced substantially to said equilibrium pressure, and thereafter removing gases desorbed from said gettering metal surface between overpressure pulses with conventional diffsion pumping means having a pump inlet adjacent said gettering metal surface.

5. The pumping method of claim 4 in which said pumping cavity communicates with a vacuum chamber and said overpressure pulses of gases are discharged and enter said pumping cavity therefrom.

6. The pumping method of claim 4 in which said gettering metal surface is renewed by feeding a wire composed of said desorbed gettering metal continuously into said pumping cavity through a coolant jacketed feed wire tube communicating through the wall thereof and disposed to position said wire vertically within line-of-sight of said gettering metal surface upon entrance therein, and bombarding the tip of said wire emerging from said tube with a beam of electrons, whereby said tip heats, melts and forms a vaporizing molten mass of metal self-supported on said tip by surface tension.

7. The pumping method of claim 4 in which said conventional pumping means is an oil diffusion pump, said pump having its inlet cooled by a cold trap whereby oil back diffusing is condensed and returned to said pump.

8. The pumping method of claim 4 in which said gettering metal is selected from the group consisting of titanium, tantalum, tungsten, molybdenum, zirconium, niobium, rhenium, thorium and uranium.

9. The pumping method of claim 4 in which said gettering metal surface comprises at least one collector plate of a Penning discharge type pump.

10. The pumping method of claim 4 in which said gettering metal is molybdenum and said gas is hydrogen and said overpressure pulses contain insufficient hydrogen to saturate said molybdenum metal surface.

11. The pumping method of claim 4 in which the ratio of gettering metal surface area to volume of gaseous pulses does not exceed the value for which said pulses can be adsorbed within one second by said surface.

12. The pumping method of claim 4 in which said overpressure pulse does not raise the pressure of the pumping cavity by a factor of more than 10 prior to adsorption.

13. A method for pumping and removing overpressure pulses of gases and gaseous particles from a vacuum cavity comprising the steps of evacuating a pumping cavity to a vacuum pressure below at least about 10^{-4} mm. Hg with a diffusion pump, pumping said cavity to an equilibrium pressure below about 10^{-6} mm. Hg by the pumping action of a degassed gettering metal selected from the group consisting of Ti, Ta, W, Mb, Zr and Nb continuously vaporized and deposited upon a gettering

metal surface within said cavity until equilibrium pumping conditions are attained, thereafter depositing additional degassed gettering metal selected from said class upon said surface only when said vacuum pressure rises above about 10^{-6} mm. Hg, adsorbing overpressure pulses which do not raise the pressure above 10^{-6} mm. Hg onto said gettering metal surface, and removing gases desorbed from said gettering metal surface between overpressure pulses when additional gettering metal is not being applied with a diffusion pump having a pump inlet adjacent said gettering metal surface, said inlet being cold trapped to prevent back diffusion of oil into said pumping cavity, whereby overpressure pulses not exceeding the adsorption capacity of said gettering metal surface are adsorbed onto said metal surface and said overpressure is reduced substantially to said equilibrium pressure.

No references cited.