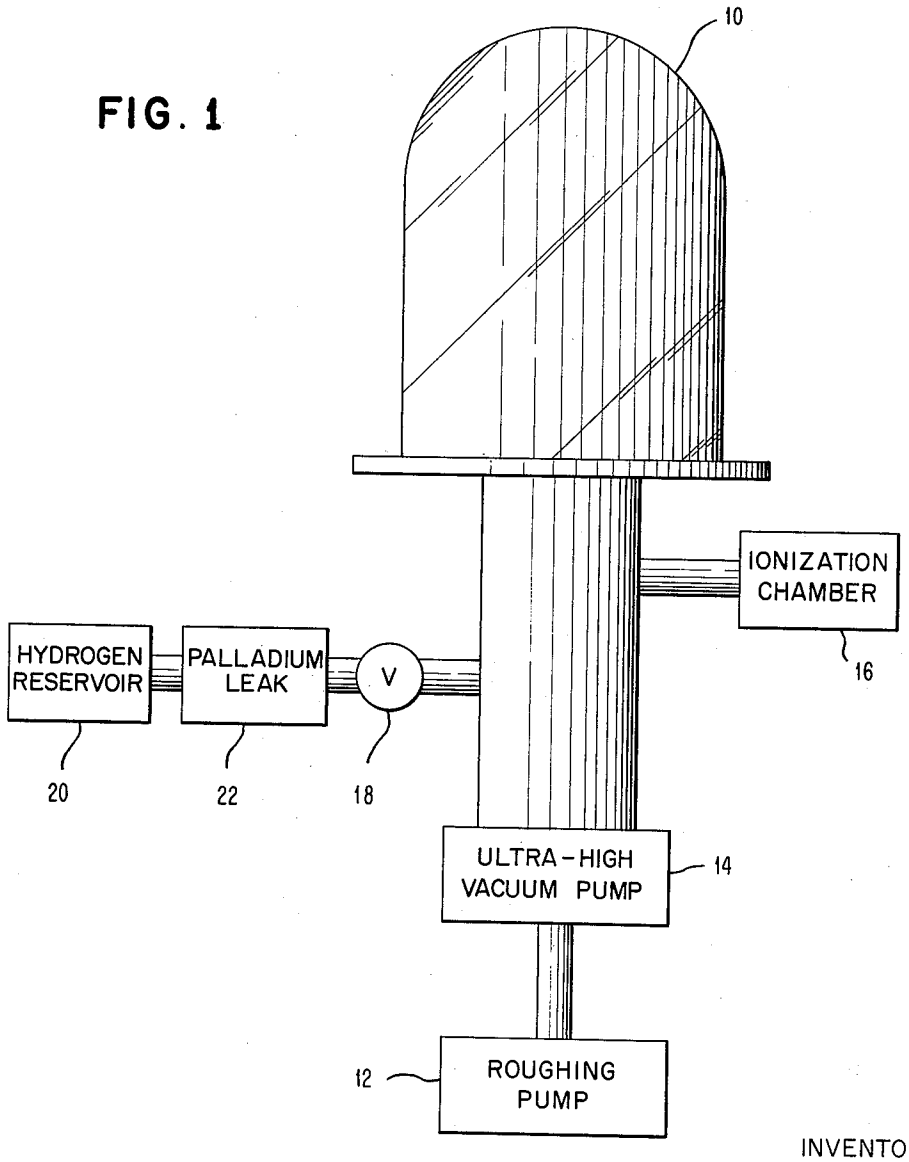
April 16, 1963 I. AMES ETAL 3,085,739

VACUUM METHOD

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FIG. 1



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FIG. 2A

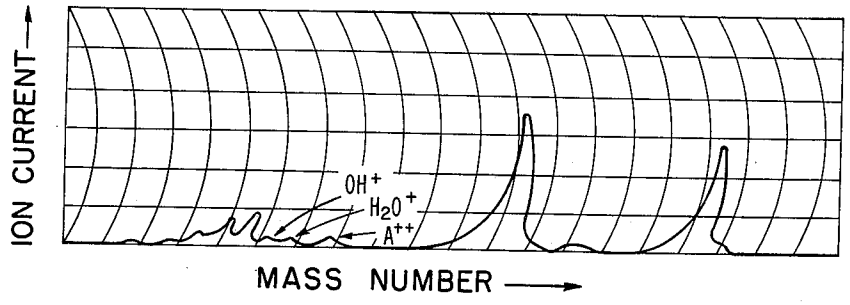


FIG. 2B

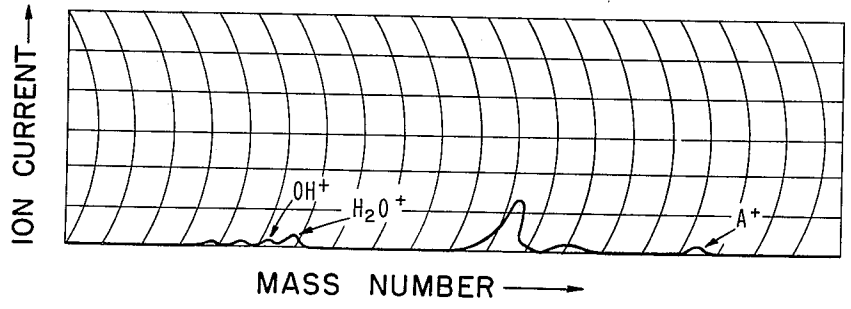
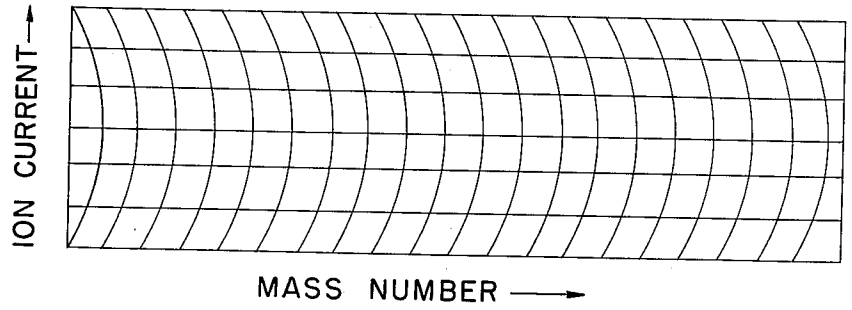


FIG. 2C



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3,085,739

VACUUM METHOD

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4 Claims. (Cl. 230—69)

This invention relates to a method of attaining a vacuum and, more particularly, to an improved method of attaining an ultra high vacuum without employing a high temperature bakeout.

In general, various methods have been employed in the prior art to evacuate vacuum systems to extremely low pressures. Additionally, various types of pumps such as, by way of example, mechanical, diffusion, and electronic pumps have all been employed to attain ultra high vacuum. Further, vacuum technology has been applied to the design of system components such as O ring seals, valves, and gauges to afford an improved vacuum system capable of attaining a lower pressure for the particular pump or pumps used. Also, to improve the limiting vacuum attainable, as determined by the system and pumps employed, the system is subjected during the pumping operation to a high temperature bakeout in order to desorb from the walls and crevices of the system molecules adhering thereto.

However, there are some systems to which, because of the materials necessarily employed therein, a high temperature bakeout cannot be applied in order to attain an increased ultra high vacuum. What has been discovered is an improvement in vacuum technology wherein pressures at least an order of magnitude lower than heretofore possible are attained in a vacuum system without employing a high temperature bakeout. Specifically, the method of this invention includes, amongst others, the steps of reducing the pressure within a vacuum system until a limiting pressure is attained which, in general, is determined by a balance between the speed of the particular pump employed and the number of residual gas molecules leaving the system walls and crevices. At this time, the pressure within the system has been reduced to a value whereat the mean free path of gas molecules within the system is determined by the system dimensions itself. Next, gaseous hydrogen at a controlled pressure which again is such that the mean free path of gas molecules within the system is determined by the system dimensions, is introduced into the chamber in the presence of an ionic discharge. The ionic discharge is effective to ionize a portion of the hydrogen, and the energetic hydrogen ions created thereby diffuse throughout the entire system to release residual gas molecules from the system walls and crevices. The hydrogen is bled into the system for a time sufficient for the vacuum pump to reduce the partial pressures of all gases within the system, except hydrogen, to a predetermined pressure. Next, the flow of hydrogen into the vacuum system is terminated and further operation of the vacuum pump is effective to attain an ultra high vacuum. This further reduction in pressure, below the limiting pressure determined by the balance between the speed of the pump employed and the number of residual gas molecules leaving the chamber walls and crevices, as a result of the introduction of the gaseous hydrogen in the presence of an ionic discharge is believed to result for the following reason. A portion of the hydrogen entering the system is ionized and, since the mean free path of gas molecules within the system is determined by the dimensions of the system itself, the energetic hydrogen ions created in the discharge diffuse throughout the chamber and are effective upon striking the walls and crevices to cause the release of one or more of the residual gas molecules adhering thereto.

The use of a working gas has hitherto been employed to reduce the pressure within vacuum systems as exemplified by U.S. Patents 1,651,386 and 2,858,972. Specifically, the latter patent is directed to lowering the pressure within a vacuum system being pumped by an ionic vacuum pump below the pressure attainable by the particular pump employed. Particularly, the ionic pump there disclosed is capable of obtaining a minimum pressure of about 10^{-6} mm. Hg, since, at pressures lower than this, the pump itself is not capable of maintaining an ionic discharge. When this limiting pressure, below which the discharge cannot be maintained, is attained, the concentration of the inert gases in the system being pumped is reduced by diluting them with a working gas which may be hydrogen, nitrogen, or oxygen to thereby increase the total pressure in the system up to a level at which the ionic discharge can be maintained, and thereafter pumping the resultant mixture at a pressure greater than the limiting pressure of the pump. An ultimate pressure below the limiting pressure of the pump is next attained by chemically gettering the residual working gas. The net result is a reduction in the total partial pressure of the inert gases in the system. It can be seen, therefore, that this final pressure is not maintainable by the pump itself, since the pump is not operable at this reduced pressure. Thus, this final pressure is merely temporary, depending on the system wherein this method is employed. However, the method of the subject invention differs markedly from the disclosures of the prior art. First, the limiting pressure prior to the introduction of hydrogen according to the method of this invention is not the limiting pressure of the pump itself. Rather, it is the limiting pressure determined by the pumping speed and the release of residual gas molecules from the surface of the unbaked system. Further, the final limiting pressure after the introduction of the hydrogen, which is rapidly attained by the method of the invention, could probably also be obtained by the pump itself without the use of hydrogen if an unreasonably long period of time, by way of example, a period of years, were available. This is in contradistinction to the method of the above briefly described patent where the limiting pressure is determined solely by the particular pump itself. Second, hydrogen, according to the method of this invention, is not introduced into the system until the pressure within the system has been reduced to a value such that the mean free path of gas molecules within the system is determined by the dimensions of the chamber itself and, further, the hydrogen is introduced at a controlled predetermined pressure at which the mean free path of the hydrogen is also determined by the dimensions of the vacuum system, to allow the beneficial cleanup action of the energetic hydrogen ions to be operable throughout the entire surface volume of the system, without the occurrence of an excess number of collisions between the generated hydrogen ions and other gas molecules within the system. Third, the limiting pressure, above referred to, results from the unbaked system itself, rather than the characteristics of the pump employed, and when the final ultra high vacuum is attained, the pump is effective to maintain the pressure within the system at this value.

An object of the invention is to provide an improved method of attaining an ultra high vacuum.

A further object of the invention is to provide a method of attaining an ultra high vacuum without employing a high temperature bakeout.

Still another object of the invention is to provide a method of attaining an ultra high vacuum wherein gaseous hydrogen is employed to complement the action of the vacuum pump.

Yet another object of the invention is to provide an improved method of attaining a vacuum wherein ion bombardment cleanup is employed.

Still another object of the invention is to provide a method of reducing the limiting pressure within an unbaked vacuum chamber.

A still further object of the invention is to provide a method of employing gaseous hydrogen and an ionic discharge to attain an ultra high vacuum.

The foregoing and other objects, features and advantages of the invention will be apparent from the following more particular descripiton of a preferred embodiment of the invention, as illustrated in the accompanying drawings.

In the drawings:

FIG. 1 is a general diagrammatic section of an apparatus for employing the method of the invention.

FIGS. 2A, 2B and 2C illustrate the reduction of the partial pressure of the various gases as a function of time within a vacuum system being pumped according to the method of the invention.

The method of the invention may be practiced whenever an ultra high vacuum is required and, further, may be practiced with any of the various vacuum systems and pumps well known in the art. FIG. 1 is a composite illustration of any of the above systems and is employed in this specification merely as an illustrative example. As is well known in the art, extreme care is necessary in selecting the various components of the system to attain an ultra high vacuum. Reference should be had to any of the well known handbooks for the selection of the materials and components forming a system as illustrated in FIG. 1. However, since the apparatus illustrated in FIG. 1 forms no part of the method of the invention, the details of the materials and components selected for use therein will not be further described, reference again being made to any of the well known vacuum handbooks.

Briefly outlined, the method of attaining an ultra high vacuum without a high temperature bakeout includes the following steps: the pressure within chamber **10** is reduced below atmospheric by means of a roughing pump indicated in FIG. 1 by block **12**, which may be, by way of example, any of the well known mechanical pumps. Since mechanical pumps are generally not capable, of and by themselves, of attaining an ultra high vacuum, it is necessary to employ a second pump. After the pressure within chamber **10** has been sufficiently reduced, by pump **12**, an ultra high vacuum pump indicated as block **14** in FIG. 1, is next operated to further reduce the pressure. By way of example, the ultra high vacuum pump may be one of the well known well-trapped mercury or oil diffusion pumps or, alternatively, an electronic or ionic pump. The operation of the ultra high vacuum pump is continued until the pumping rate thereof is essentially equal to the rate at which sorbed gas molecules re-enter the chamber volume from the surfaces and crevices therein. Since an ultra high vacuum pump is employed in a well designed vacuum system, it should be understood that this first limiting pressure is reduced to a pressure such that the mean free path of gas molecules within chamber **10** is determined solely by the dimensions of the vacuum chamber itself. Next, an ionic discharge is obtained within the system, which may be, by way of example, a device generally indicated as **16** in FIG. 1, which includes a directed beam of electrons. At this time, gaseous hydrogen is introduced into the system at a controlled and predetermined pressure. This may be accomplished, as indicated in FIG. 1, by the operation of a valve **18** connected to a hydrogen reservoir **20** through a palladium leak **22**. This introduction of hydrogen and the ionic discharge is effective to generate energetic hydrogen ions which are further effective to clean up the interior surfaces of chamber **10** whereby the partial pressure of residual gas molecules, other than hydrogen, remaining in the chamber are reduced by pump **14**.

When each of these partial pressures has been reduced below a predetermined value, valve **18** is closed and further pumping by pump **14** is effective to attain an ultra high vacuum, which is at least an order of magnitude below the limiting pressure as determined by the balance between the speed of pump **14** and the number of residual gas molecules leaving the chamber walls and crevices. As a particular example, the following more detailed description of the method is next described. The specific pressures and times shown therein are by way of illustration only, it being understood that a wide departure from these pressures and times may result from the particular systems and pumps employed without departing from the scope of the present invention.

Further, by way of illustration, the method of the invention is described employing an ion-getter pump. The use of this pump is effective simultaneously to both reduce the pressure within chamber **10** and to function as the necessary ionization chamber for generating the energetic hydrogen ions, ionization chamber **16**, therefore, not being necessary in this example. After the chamber has been vacuum sealed, pump **12** is operated for a relatively short time, in the order of 10 minutes to reduce the pressure within the chamber from atmospheric to the range 10^{-2} to 10^{-3} mm. Hg, and is then sealed from the system by an internal isolation valve. At this time, the ultra high vacuum pump **14**, which as stated above is an ion-getter pump by way of example, is placed in operation to further reduce the pressure within chamber **10**. Continued pumping by pump **14** is effective to reduce the pressure to about 10^{-8} mm. Hg. At this pressure, a further reduction in the pressure in chamber **10** is not attainable due to the quantity of sorbed gas molecules adhering to the surfaces of the vacuum chamber and, as hereinbefore described, the release of these molecules generally prevents the pressure within chamber **10** from dropping below the order of 10^{-7} to 10^{-8} mm. Hg. At this time, valve **18** is opened to allow gaseous hydrogen from reservoir **20** to enter chamber **10** at a controlled and predetermined pressure of about 10^{-5} mm. Hg. A portion of the hydrogen is ionized by pump **14**, and since the mean free path, at the low pressure in the system of the energetic ions created thereby is determined solely by the dimensions of the system, these ions are effective to clean up the entire interior surfaces and crevices of chamber **10**, pump **14** then being effective to reduce the partial pressures of the residual molecules of the gases other than hydrogen remaining within the chamber to less than 10^{-9} mm. Hg. At this time, valve **18** is reclosed and continued pumping by pump **14** is effective to reduce the partial pressure of hydrogen, and, hence, the total pressure within the chamber, to about 10^{-9} mm. Hg. Through this combination of steps, therefore, an ultra high vacuum is attained and maintained without employing a high temperature bakeout. As an aid in understanding the action of the hydrogen gas cooperating wtih discharge **16** or, alternatively, when an ion-getter pump is employed, with pump **14**, reference should now be had to FIGS. 2A, 2B and 2C, which indicate the partial pressures of the various gases within chamber **10**, as measured by a mass spectrometer during the specific example described immediately above. As shown, the ordinate in each of FIGS. 2A, 2B, and 2C indicates the number of ions of equal mass to charge ratio (m/e), or mass number, as measured by the mass spectrometer, several of which, being unique, are identified. In addition to the partial pressures of the various gases as indicated in each of these figures, additional peaks resulting from H_1^+, H_2^+ and H_3^+ are observed, but in the interest of clarity are not indicated in these figures. However, the peak height of the partial pressure of H_3^+ is proportional to the product of H_1^+ and H_2^+ indicating that the H_3 ions are generated by collision reactions between the H_1 and H_2 ions. FIG. 2A indicates the partial pressure of the gases within chamber **10** several hours after the introduction

of the gaseous hydrogen, the major peak being a combination of N_2^+, CO^+, and $C_2H_4^+$ ions, all of which have equal mass to charge ratios. FIG. 2B indicates the further reduction in the partial pressures of the residual gases within chamber **10** after an additional several hours of pumping by pump **14** with the pressure of hydrogen maintained at a level of about 10^{-5} mm. Hg. As shown in FIG. 2C, after several additional hours of pumping in the presence of hydrogen, no residual gases are indicated. Since the sensitivity of the mass spectrometer employed was limited to a partial pressure of about 10^{-9} mm. Hg, FIG. 2C indicates that the partial pressures of all gases, except, of course, hydrogen which is maintained at about 10^{-5}, had been reduced below 10^{-9} mm. Hg. At this time, valve **18** is reclosed terminating the supply of hydrogen to chamber **10** and several additional hours of pumping by pump **14** results in a total pressure of about 10^{-9} being attained. Note should be made of the fact that the location of ionization chamber **16**, or alternatively, ion-getter pump **14** with respect to work chamber **10** is relatively immaterial. This occurs since, as has been emphasized above, the mean free path of hydrogen within the system is determined by the system dimensions. Thus, a portion of the hydrogen is ionized in the discharge and the energetic ions created thereby diffuse throughout the system unimpeded by collisions with other gas molecules.

Although the steps described in detail immediately above results in obtaining an ultra high vacuum in the range of about 10^{-9} mm. Hg, the method of the invention may also be employed to attain pressures significantly higher when the ultimate in pressure is not desired. It should be understood, however, that the hydrogen should not be introduced into the system until the pressure therein has been reduced to a point where the mean free path of the hydrogen atoms is determined solely by the dimensions of the vacuum system itself.

This reduction in system pressure, as a result of the introduction of hydrogen is produced in the following manner. First, the mean free path of hydrogen at a pressure in the range of 10^{-3} mm. Hg is about 5,000 meters and at a pressure of 10^{-5} mm. Hg is about 5 meters. Thus, the effective mean free path for the hydrogen within chamber **10** can be considered to be determined by and limited to the dimensions of the chamber itself. A portion of the hydrogen entering the system is ionized by ionization chamber **16** or ion-getter pump **12** and the generated ions, therefore, diffuse throughout the entire system and have a high probability of interacting with one or more of the sorbed gas molecules on the walls of the system, prior to collisions with one or more gas molecules in the volume of the system. The energetic ions thus retain their energy until a portion thereof is transferred to the sorbed gas molecules, thus causing their release from the walls and crevices of the system. Second, when an ion-getter pump is employed, the introduction of hydrogen is effective to cause an excess amount of getter material to be deposited within the pump over that which would normally be deposited at the pressure prior to the hydrogen introduction. This deposition of excess getter material may then provide added pumping capabilities for the other gases within chamber **10**.

What has been described is a novel approach to vacuum technique wherein the final pressure obtainable within an unbaked system approaches that obtainable in a baked system. The novel cooperation of hydrogen together with an ionization chamber or ion-getter pump produces ionic and chemical cleanup of the system resulting in pressures being obtained in the unbaked system lower than that previously obtainable.

While the invention has been particularly shown and described with reference to a preferred embodiment thereof, it will be understood by those skilled in the art that various changes in form and details may be made therein without departing from the spirit and scope of the invention.

In the claims:

1. The method of attaining and maintaining an ultra high vacuum within a vacuum system without employing a high temperature bakeout comprising the steps of; reducing the pressure within said system with an ultra high vacuum pump until a first limiting pressure is attained; said first limiting pressure being determined by the pumping speed of said pump and the number of gas molecules desorbing from the interior surfaces of said system; introducing a flow of hydrogen at a predetermined pressure into said system; ionizing a portion of said hydrogen, said hydrogen ions being thereafter effective to increase the rate of desorption of gas molecules from said surfaces of said systems; reducing the partial pressure of each of said gas molecules to a second limiting pressure less than said first limiting pressure by said ultra high vacuum pump; terminating said flow of hydrogen; and further reducing the total pressure within said system to said second limiting pressure by said ultra high vacuum pump to thereby attain an ultra high vacuum, said ultra high vcauum pump, of and by itself, thereafter maintaining said ultra high vacuum.

2. The method of attaining and maintaining an ultra high vacuum within a vacuum system without employing a high temperature bakeout comprising the steps of; operating an ultra high vacuum pump to reduce the pressure within said system to a first value; said first value being determined both by the pumping rate of said pump and the rate of desorption of residual gas molecules within said system; introducing into said system a flow of hydrogen gas at a second value of pressure; the sum of said first and second pressure values resulting in a third value of pressure at which the mean free path of gas molecules within said system is determined by the system dimensions only; maintaining an ionic discharge within said system; said discharge effective to generate energetic hydrogen ions; said energetic hydrogen ions thereafter effective to increase said rate of desorption of residual gas molecules throughout said entire system; maintaining said flow of hydrogen gas for a time sufficient to reduce the partial pressure of said residual gas molecules to a fourth value of pressure; said fourth value being less than said first value of pressure; terminating the flow of hydrogen and said discharge; and operating said pump to attain and maintain an ultra high vacuum.

3. The method of attaining an ultra high vacuum within an unbaked vacuum system having residual gas molecules adhering to the surfaces thereof, which method comprises; operating a roughing pump to reduce the pressure within said system from atmospheric to about 10^{-2} mm. Hg; operating an ion-getter pump to further reduce the pressure within said system to about 10^{-7} mm. Hg; introducing a flow of hydrogen at a controlled pressure of about 10^{-5} mm. Hg into said system, whereby a portion of said hydrogen is ionized by said ion-getter pump; said generated hydrogen ions effective to release said residual gas molecules from said system surfaces; continuing the operation of said ion-getter pump and said flow of hydrogen for a time sufficient to reduce the partial pressure of each residual gas to less than 10^{-9} mm. Hg; terminating the flow of hydrogen into said system; and further operating said ion-getter pump for a time sufficient to attain an ultra high vacuum.

4. The method of attaining an ultra high vacuum within an unbaked vacuum system comprising; operating an ultra high vacuum pump to reduce the pressure within said system to a first limiting pressure; said first limiting pressure being determined by the balance between the pumping speed of said pump and the rate at which sorbed gas molecules are released from the interior surfaces of said system; introducing a flow of hydrogen at a controlled and predetermined pressure greater than said first pressure into said system; said predetermined pressure

resulting in the mean free path of gas molecules within said system being determined by the dimensions of said system; generating an ionic discharge within said system, said discharge effective to generate energetic hydrogen ions; maintaining said flow of hydrogen and said ionic discharge for a time sufficient for said pump to reduce the partial pressures of the residual gases within said system below a second limiting pressure; terminating said flow of hydrogen and said ionic discharge; and further operating said pump until the total pressure within said system is substantially equal to said second limiting pressure.

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