United States Patent

[11] 3,609,064

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[54] **GETTER PUMP WITH DIRECT RESISTANCE HEATING OF GETTER STRIP**
11 Claims, 2 Drawing Figs.

[52] **U.S. Cl.** **417/51,** 417/53, 55/208, 55/387
[51] **Int. Cl.** **F04b 37/02,** B01d 53/26, B01d 39/00
[50] **Field of Search** 417/48, 49, 51, 53; 55/208, 387, 389

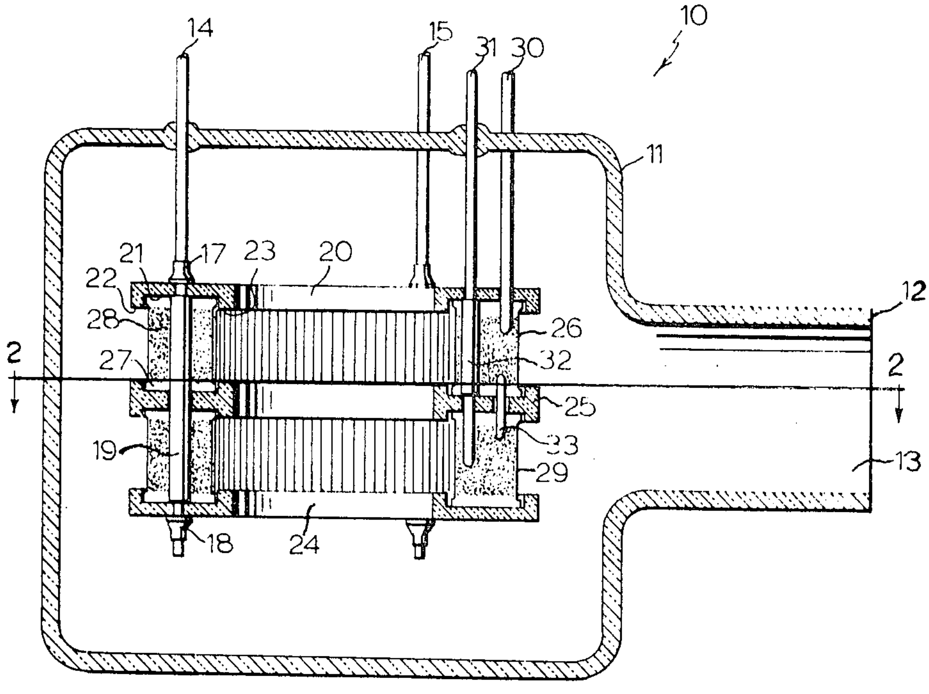
[56] **References Cited**

UNITED STATES PATENTS

2,965,218	12/1960	Jayne	417/51
3,469,375	9/1969	Barrington et al.	55/208

Primary Examiner—Robert M. Walker
Attorney—David R. Murphy

ABSTRACT: A getter pump comprising a substrate of high ohmic resistance; a nonevaporable getter material embedded in the substrate; and means for causing an electrical current to flow through the substrate.



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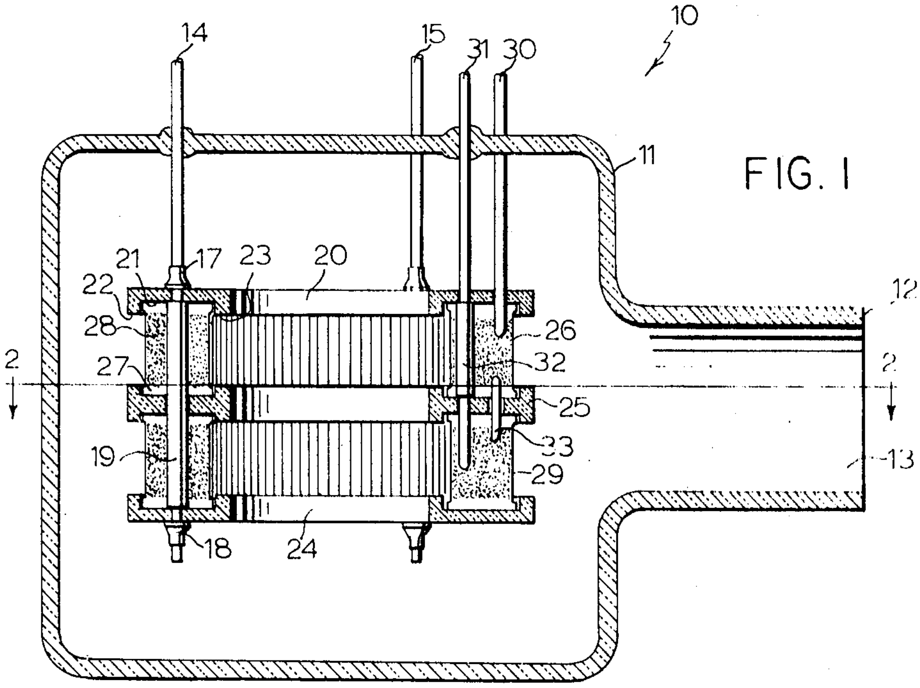
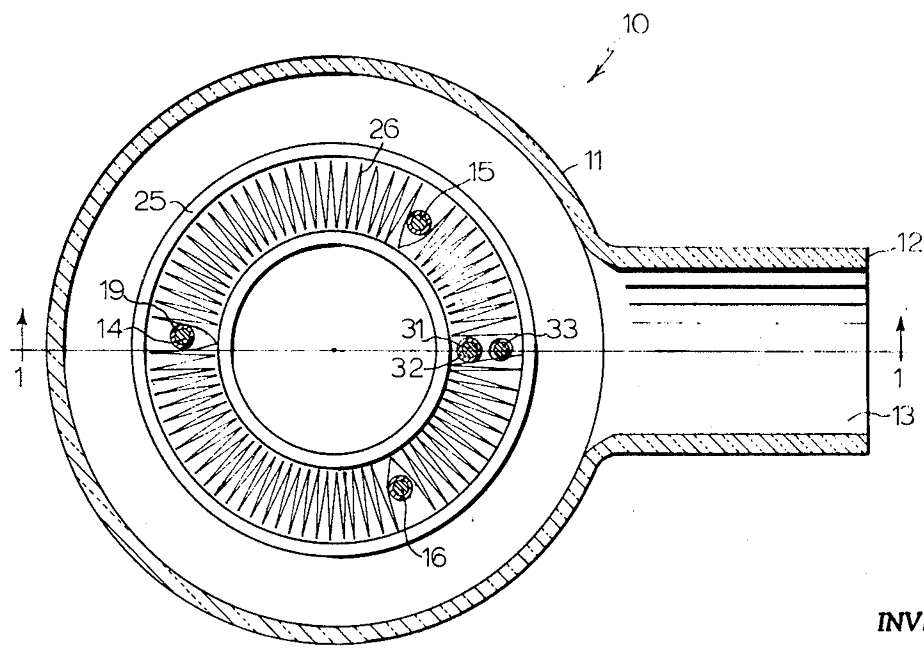


FIG. 1

FIG. 2



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GETTER PUMP WITH DIRECT RESISTANCE HEATING OF GETTER STRIP

Getter pumps employing nonevaporable getter materials embedded in a substrate are known and have found wide acceptance for producing and maintaining a vacuum is closed vessels and especially in electronic tubes such as klystron tubes, image intensifier tubes and cathode-ray tubes. Such pumps employing getter materials are especially suited for the pumping of active gases. The substrate coated with the getter material is generally pleated and coaxially disposed around a separate central resistance heater. In operation heat is radiated from the resistance heater to the getter material on the substrate. If the getter material has a clean fresh surface the sorptive rate of gases increases with temperature as is well known in the art. If the surface of the getter material is partially or fully saturated with sorbed gases, the heat drives the gas-reaction products, through diffusion, into the center of the particles of the getter material and exposes a fresh surface. Unfortunately such devices suffer from a number of disadvantages. The presence of the necessary separate resistance heater increases production costs. Uniform heating of the getter material is difficult or impossible to obtain for example, because varying portions of the coated substrate are at varying distances from the heater. While it is desirable to coat both sides of the substrate with the getter material only the side next to the heater is heated by radiation whereas the side away from the heater must be heated by conduction through the substrate. Because of the above, relatively long heating periods are required in order to activate the getter material.

Accordingly, it is an object of the present invention to provide getter pumps which are substantially free of one or more of the disadvantages of prior pumps.

Another object is to provide getter pumps which require no separate heater.

A further object is to provide getter pumps wherein the getter material can be easily and uniformly heated.

A still further object is to provide getter pumps employing a substrate having a getter material on both sides which is heated uniformly and simultaneously.

Still another object is to provide a getter pump which requires only a short period of time to activate the getter material.

Still another object is to provide a novel process for activating a nonevaporable getter material which is embedded in a substrate.

Additional objects and advantages of the present invention will be apparent to those skilled in the art by reference to the following detailed description and drawings wherein:

FIG. 1 is a sectional view of a getter pump of the present invention taken along line 1—1 of FIG. 2; and,

FIG. 2 is a sectional view of a getter pump of the present invention taken along line 2—2 of FIG. 1.

The above and other objects are accomplished by providing a getter pump having a nonevaporable getter material embedded in a substrate of high ohmic resistance and means for causing an electrical current to flow through the substrate to heat the getter material.

Referring now to the drawings and in particular to FIG. 1 there is shown a nonlimiting preferred embodiment of the present invention in the form of a getter pump 10 comprising a glass, gastight envelope 11 having a rim 12 which defines an opening 13. By means of the rim 12 the getter pump 10 is rendered attachable to any vessel in which it is desired to maintain a vacuum. Fixedly attached to and extending through the envelope 10 are three support members 14, 15 and 16 (See FIG. 2). The support member 14 has near the end nearest to the envelope 11 a first flanged washer 17 electrically spot welded to the support member 14 and acting as a stop. Near the end of the support member 14 which is furthest from the point of attachment of the support member 14 and the envelope 11 is a second flanged washer 18. A cylindrical sleeve 19 of heat and electrical insulative material surrounds the portion of the support 14 between the first flanged washer 17 and the second flanged washer 18. As shown in FIGS. 1 and 2 the support members 15 and 16 have similar flanged washers and sleeves. Within the envelope 11 is a first retainer 20 having a hole through which the support member 14 passes, the diameter of the hold being less than the diameter of the sleeve 19 such that the retainer 20 is fixedly clamped on the support 14 between the first flanged washer 17 and the sleeve 19. The retainer 20 has a flat portion 21 attached to an outer rim 22 and an inner rim 23. The flat portion 21 in the inner and outer rims 22 and 23 together form an annular groove. Within the envelope 11 is a second retainer 24 identical to the first retainer 20 but with its annular groove facing the annular groove of the retainer 20. The second retainer 24 is fixedly held on the support member 14 by the second flanged washer 18 and the sleeve 19. Slidably mounted on the sleeve 19 is an intermediate retainer 25 having annular grooves on opposite faces. Between adjacent retainers 20 and 25 is a pleated strip 26 comprising a substrate 27 having thereon a particulate nonevaporable getter material 28. A similar pleated strip 29 is between the second retainer 24 and the intermediate retainer 25.

A first electrode 30 extends through the envelope 11 and is electrically spot welded to the pleated strip 26 forming an electrical contact therewith. Also extending through the envelope 11 is a second electrode 31 which is likewise spot welded to the pleated strip 29. Surrounding the second electrode 31 in that portion of the electrode between the first retainer 20 and the intermediate retainer 25 is an insulative sleeve 32. As shown in FIG. 2 conductor 33 connects the end of the pleated strip 26 with the beginning of the pleated strip 29. Thus the pleated strips 26 and 29 comprise two electrical resistances connected in series with electrodes 30 and 31. The strips 26 and 29 can be connected in parallel but series is the preferred arrangement because of the lower current required for equivalent heating. The retainers 20, 24 and 25; and the sleeves 19 and 32 can be of any heat resistant dielectric material but are preferably ceramic.

The substrate 27 can be any material of high ohmic resistance such that passage of current therethrough heats the getter material 28 to within the desired temperature range. However the substrate 27 is generally formed of a material having a resistivity of 1 to 200 and preferably 10 to 150 microhm-centimeters when measured at 20° C. Within these limits of resistivity practical current values can be employed. Examples of suitable substrate materials include among others stainless steel containing 18 degrees chromium and 8 degrees nickel, balance consisting essentially of iron; as well as the widely used high resistance material available under the trade name "Nichrome." Other suitable materials will immediately be apparent to those skilled in the art.

In the broadest aspects of the present invention any nonevaporable getter material can be employed such as titanium, zirconium, tantalum or niobium as well as alloys and/or mixtures of two or more of the above. The preferred nonevaporable getter material is an alloy of 5 to 30 weight percent aluminum, balance zirconium, and especially that alloy of 16 weight percent aluminum, balance zirconium, available as "St 101" from S.A.E.S. Getters S.p.A., Milan, Italy.

The getter material 28 is applied to the substrate 27 in the form of a powder in order to have a high surface area to mass ratio facilitating gas sorption. The powder is preferably one which passes through a U.S. Standard screen of 140 mesh/inch. The powder is attached to the strips 26 and 29 by any suitable means such as rolling or pressing which does not materially reduce the total surface area of the powder.

In operation the getter pump 10 is attached to a vessel to be evacuated by sealing the rim 12 to the vessel. The vessel is then evacuated by any convenient means such as a mechanical pump or a zeolite pump. Then the electrodes 30 and 31 are connected to a source of alternating or direct current whereby current flows through the pleated strip 26 and the pleated strip 29 activating the getter material, ohmically generating heat in the substrate 27, conducting the heat to the particulate nonevaporable getter material 28 and driving the sorbed gases into the interior of each particle of getter material. Current is passed through the strips 26 and 29 such that the temperature of the getter material 28 is held at 600° to 900° and preferably 700° to 800° C. At temperatures below this range activation is too slow to be practical whereas at temperatures above this range sinterization of the particles of the getter material 28 begins to occur together with diffusion of the metal of the strips 26 and 29 into the getter material 28 and vice versa. Both sinterization and diffusion tend to reduce the gas sorptive capacity of the getter material 28.

Once activation is accomplished the getter material 28 is gas sorptive at room temperature but the rate of gas sorption can be increased by heating the getter material 28 as described above or more preferably at temperatures of 250° to 400° C. in order to avoid the evolution of hydrogen, which can be present in the getter material 28 as a solid solution due to previous hydrogen sorption. The getter material 28 remains gas sorptive after heating is terminated and continues to sorb active gases evolved during the life of the vessel. Should an undesirable increase in gas pressure in the vessel occur, it is only necessary to connect the electrodes 30 and 31 to a source of power in order to reactivate the getter material 28. In this manner, a very high vacuum can be maintained in a vessel throughout its life and until the getter material 28 becomes saturated with gases.

The getter pumps of the present invention find utility as supplements to sputter ion pumps and diffusion pumps and can be used to create and maintain vacuum in continuously pumped vacuum systems and in sealed off vacuum systems. These pumps can be permanently installed for example in klystron tubes and image intensifier tubes as so called appendage pumps.

The invention is further illustrated by the following nonlimiting examples which are designed to illustrate the best mode of the present invention.

Example I

This example illustrates the construction of a substrate having embedded therein a nonevaporable getter material which substrate is useful in the present invention.

Finely ground St101 nonevaporable getter material is passed through as screen having 140 mesh/inch and is retained on a screen having 600 mesh/inch. This material is then placed on the center of a strip of 18–8 stainless steel 8 mm. wide and 0.2 mm. thick leaving a getter material free margin near the edges of the strip. On top of the powder is placed a strip of common soft iron of the same dimensions. The two strips with the St101 alloy between them are then pressed together by a roller whereupon the strip of soft iron is removed leaving the St101 alloy adhering to the strip of stainless steel. A plurality of slits are then made at intervals of 6 mm. across the strip and extending into the margin which is free of St101 alloy. The strip is then folded adjacent to the slits to form a pleated strip which is then circularly curved to be employed in the getter pump of the present invention.

EXAMPLE II

A substrate having a getter material embedded thereon is produced as described in U.S. application Ser. No. 527,906, filed Feb. 16, 1966. The substrate is then pleated as in example I.

EXAMPLE III

A substrate coated on both sides with St101 alloy is purchased from S.A.E.S. Getters S.p.A. of Milan, Italy, under stock number St1011Ct/86/D60 and pleated as described in example I.

EXAMPLE IV

A substrate coated on both sides with St101 alloy is purchased from S.A.E.S. Getters S.p.A. of Milan, Italy under stock number St101/Ct/15×6/D150.

EXAMPLE V

The pleated strip of example III is circularly formed as shown in FIGS. 1 and 2, attached to electrodes 30 and 31 and placed in a getter pump 10 as shown in FIGS. 1 and 2. The getter pump 10 is then attached to a glass vessel of about 1 liter in volume whose pressure is reduced to 10^{13} torr using zeolites whereupon a current of 7 amps is passed through the electrodes 30 and 31 for a period of 10 minutes activating the getter material and reducing the pressure of active gases in the vessel to 10^{18} torr in an overall time of 20 minutes. Similar results are obtained with the pleated strips of examples I, II and IV.

Although the invention has been described in considerable detail with reference to certain preferred embodiments thereof, it will be understood that variations and modifications can be effected within the spirit and scope of the invention as described above and as defined in the appended claims.

What is claimed is:

1. A getter pump comprising a substrate of high ohmic resistance; a particulate nonevaporable getter material embedded in the substrate; and means for causing an electrical current to flow through the substrate wherein the substrate is of a material having a resistivity of 1 to 200 microhm-centimeters when measured at 20° C.

2. The pump of claim 1 wherein the nonevaporable getter material is an alloy of 5 to 30 weigh percent aluminum, balance zirconium.

3. The pump of claim 2 wherein the nonevaporable getter material is an alloy of 16 weight percent aluminum, balance zirconium.

4. The pump of claim 1 wherein the means for causing an electrical current to flow comprises electrodes attached to opposite ends of the substrate.

5. The pump of claim 1 wherein the substrate is pleated.

6. The pump of claim 5 wherein the pleated substrate is circularly formed.

7. A getter pump of claim 1 comprising:

A. a gastight envelope having a rim defining an opening attachable to a vessel in fluid communication therewith;

B. a first retainer fixed attached to the envelope, the first retainer having an annular grooves;

C. a second retainer fixedly attached to to envelope, the second retainer having an annular groove facing the annular groove of the first retainer;

D. at least one strip of planar substrate of a material of high ohmic resistance having embedded in the substrate a particulate nonevaporable getter material, the pleated strip being between the retainers resting in the annular grooves of adjacent retains; and,

E. means for causing an electrical current to flow through the substrate, whereby imposing a potential across the electrodes causes current to flow through the pleated strip heating and activating the embedded getter material thereby increasing the rate of sorption of gas within the vessel.

8. A getter pump comprising a substrate of high ohmic resistance; a particulate nonevaporable getter material embedded in the substrate; and means for causing an electrical current to flow through the substrate wherein the substrate is of a material having a resistivity of 10 to 150 microhm-centimeters when measured at 20° C.

9. A process for increasing the sorptive rate of particulate nonevaporable getter material embedded in a substrate comprising the step of passing an electrical current through the substrate thereby ohmically generating heat in the substrate and conducting the heat to the particulate nonevaporable getter material wherein the electrical current is passed at a

rate such that the particulate nonevaporable getter material is heated to 600° to 900° C. to activate the getter material.

10. A process for increasing the sorptive rate of a particulate nonevaporable getter material embedded in a substrate comprising the step of passing an electrical current through the substrate thereby ohmically generating heat in the substrate and conducting the heat to the particulate nonevaporable getter material wherein the electrical current is passed at a rate such that the particulate nonevaporable getter material is maintained at a temperature of 250° to 400° C. in order to sorb active gases at a maximum rate while avoiding evolution of hydrogen.

11. A getter pump comprising a substrate of high ohmic resistance; a particulate nonevaporable getter metal embedded in the substrate and means for imposing a potential across the substrate in order to cause an electrical current to flow through the substrate heating it and conducting the heat to the particulate nonevaporable getter metal embedded therein.

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,609,064 Dated September 28, 1971

Inventor(s) Tiziano A. Giorgi et al

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Col 1, Line 5, delete "is" and insert --in--.

Col 2, Line 4, delete "hold" and insert --hole--.

Col 2, Line 46, delete "degrees" and insert --percent-- (both occurrences)

Col 3, Example III, Line 74, delete "St1011Ct/86/D60" and insert --St 101/Ct/8 x 6/D60--.

Col 4, Line 12, delete "10^{13}" and insert --10^{-3}--.

Col 4, Line 16, delete "10^{18}" and insert --10^{-8}--.

Col 4, Line 44, delete "of Claim 1".

Col 4, Line 47, delete "fixed" and insert --fixedly--.

Col 4, Line 48, delete "grooves" and insert --groove--.

Col 4, Line 49, delete "to" (second occurrence) and insert --the--

Col 4, Line 57, delete "retains" and insert --retainers--.

Signed and sealed this 9th day of May 1972.

(SEAL)
Attest:

EDWARD M.FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents