United States Patent [19]

Barosi

[11] **3,926,832**

[45] **Dec. 16, 1975**

[54] **GETTERING STRUCTURE**

[75] Inventor: **Aldo Barosi,** Milan, Italy

[73] Assignee: **S.A.E.S. Getters S.p.A.,** Milan, Italy

[22] Filed: **July 30, 1973**

[21] Appl. No.: **383,677**

[30] **Foreign Application Priority Data**

Aug. 10, 1972 Italy 28053/72

[52] **U.S. Cl.**................ **252/181.6;** 75/.5 K; 29/191.2

[51] **Int. Cl.**........ **H01j 7/18;** H01j 35/20; H01j 1/50

[58] **Field of Search**.............. 252/181.1, 181.6, 464, 252/517, 518, 520; 75/.5 BB, 177; 29/182, 191.2

[56] **References Cited**

UNITED STATES PATENTS

2,855,368	10/1958	Perdijk et al.	252/181.6
2,966,736	1/1961	Towner et al.	29/182
2,967,351	1/1961	Roberts et al.	29/182
3,203,901	8/1965	Della Porta	252/181.6
3,584,253	7/1971	Wintzer	252/181.6
3,652,317	3/1972	Della Porta	117/22
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404,129	1965	Japan	29/182
1,302,951	1971	Germany	252/181.6
1,011,259	1965	United Kingdom	252/181.6

Primary Examiner—Jack Cooper
Assistant Examiner—Eugene T. Wheelock
Attorney, Agent, or Firm—Littlepage, Quaintance, Murphy & Dobyns

[57] **ABSTRACT**

A non-evaporating getter device and composition and process for producing such which is optionally heatable employing a getter material comprising at least one non-evaporable getter metal preferably selected from the group consisting of Zr, Ta, Hf, Nb, Ti, Th and U in intimate mixture with a zirconium-aluminum alloy.

5 Claims, 11 Drawing Figures

FIG. 1

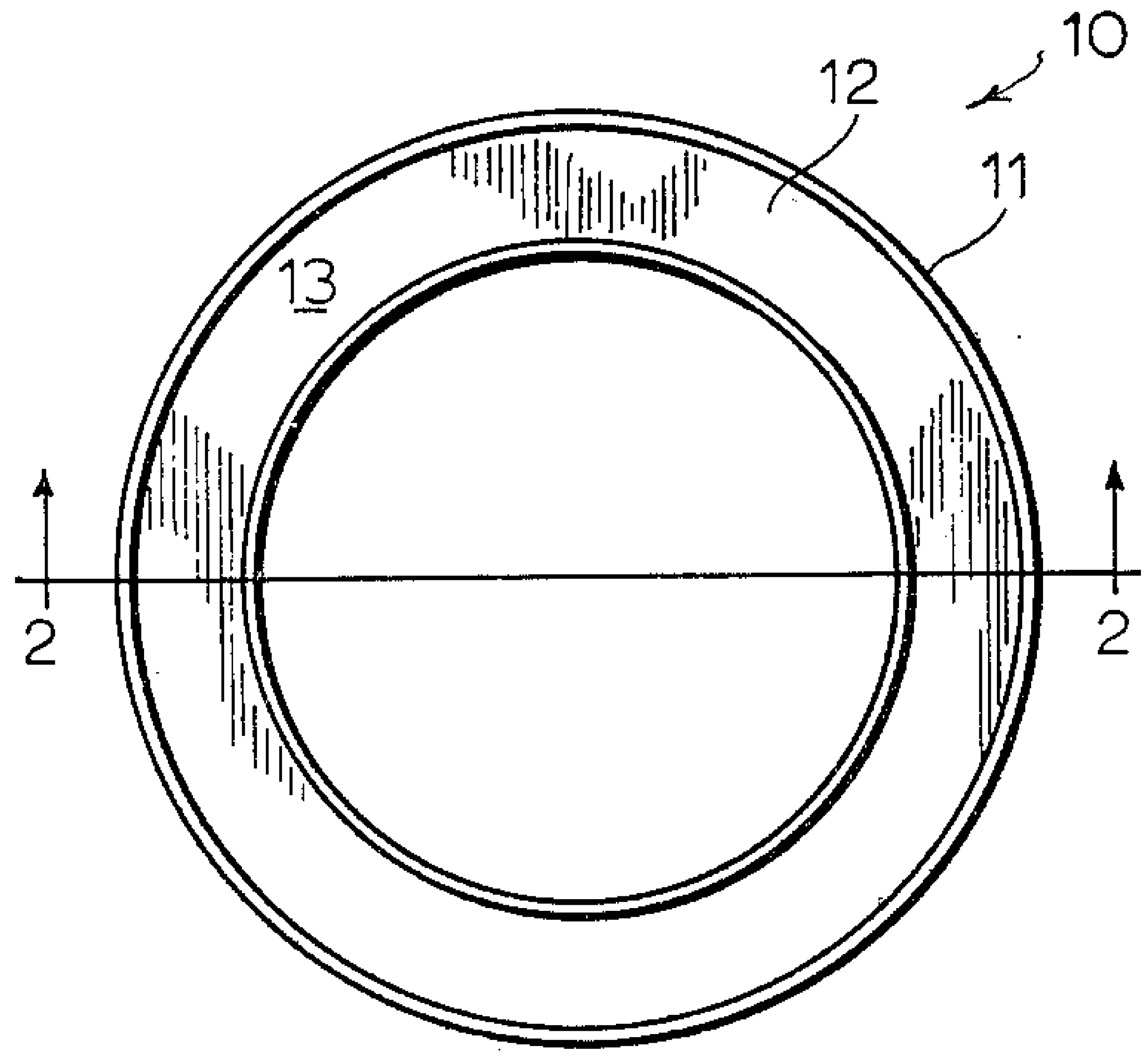


FIG. 7

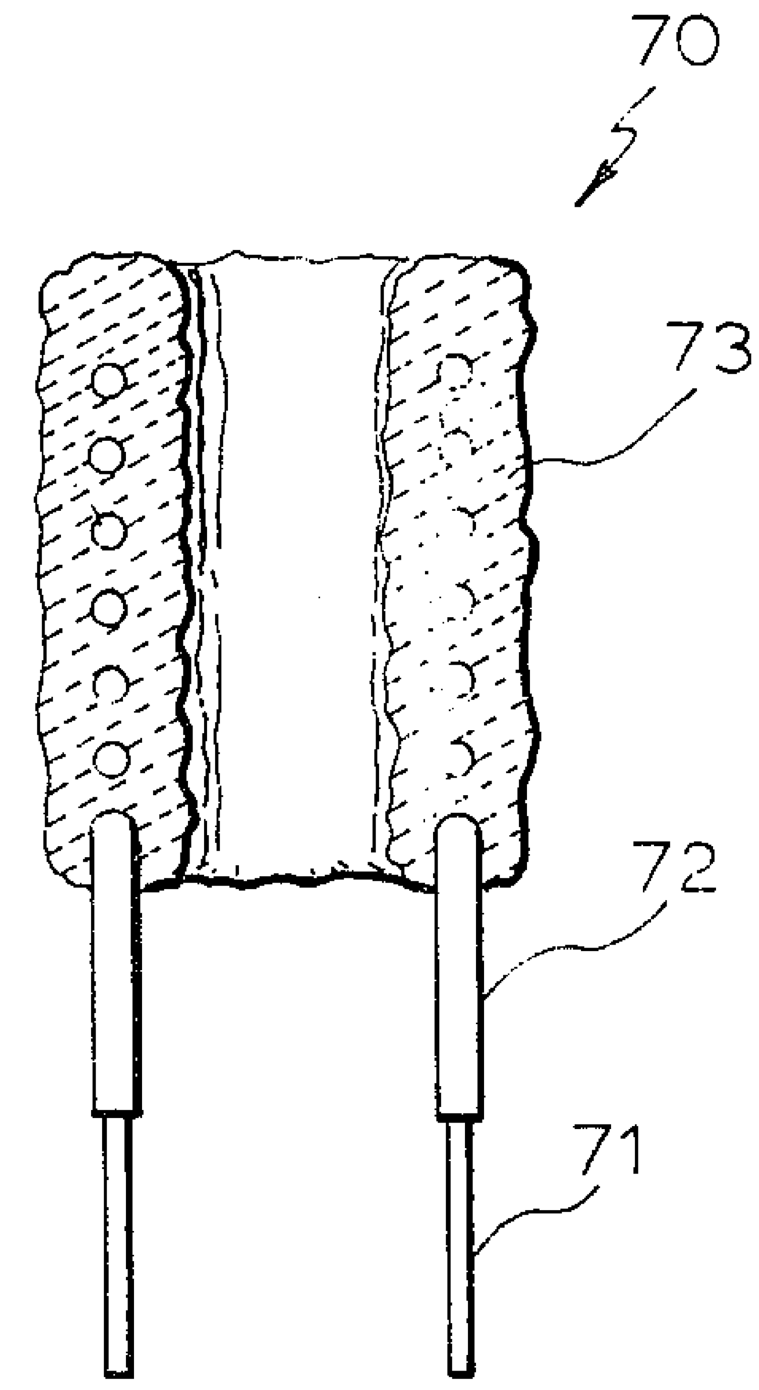


FIG. 2

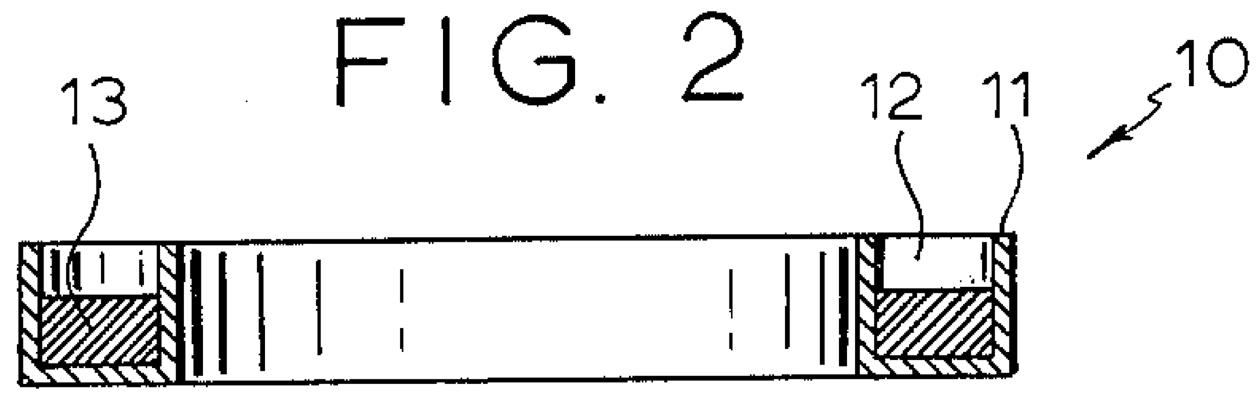


FIG. 3

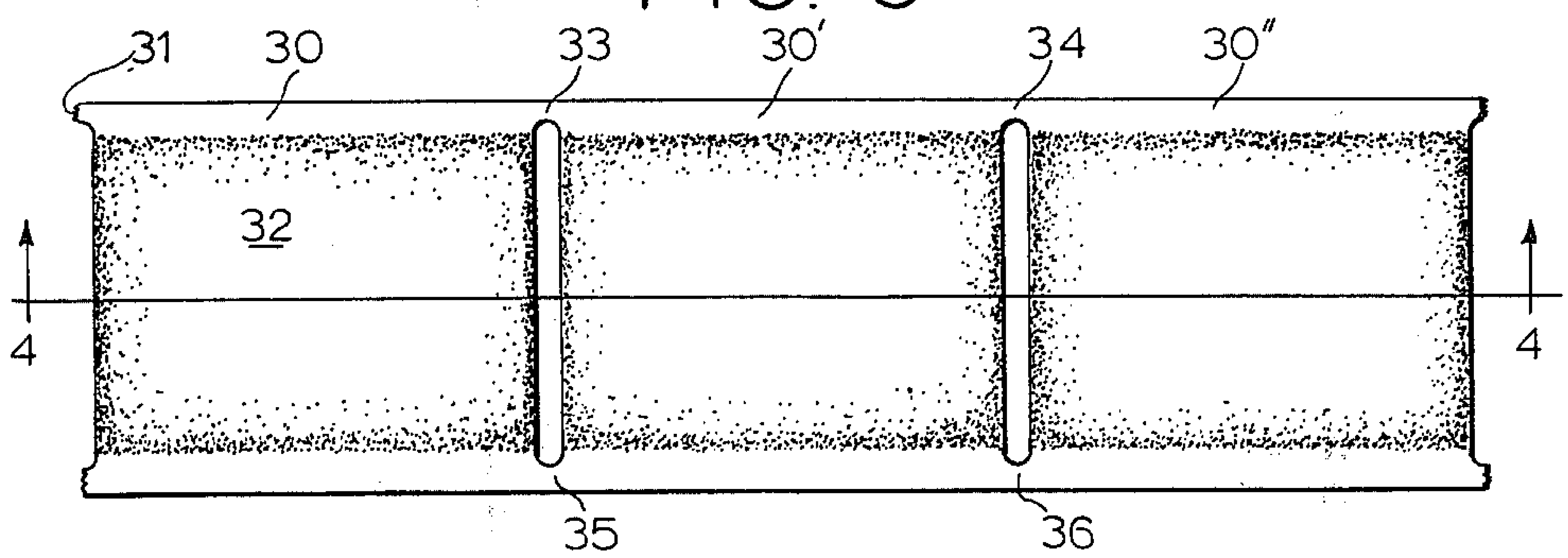


FIG. 4

FIG. 5

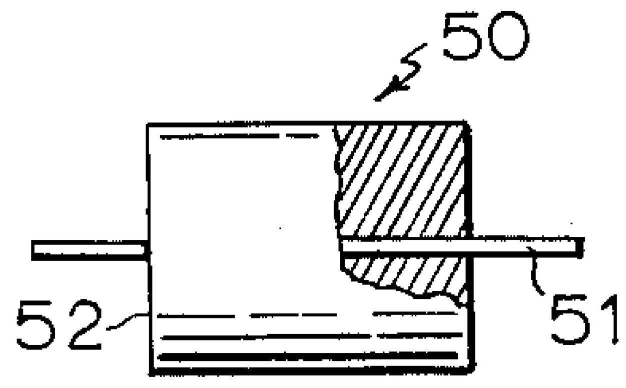


FIG. 6

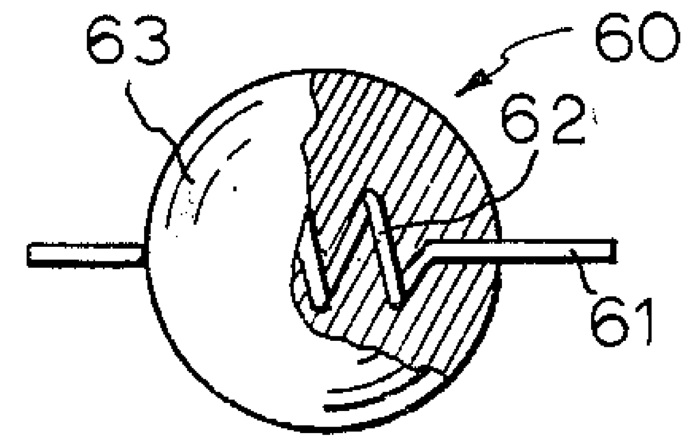


FIG. 8

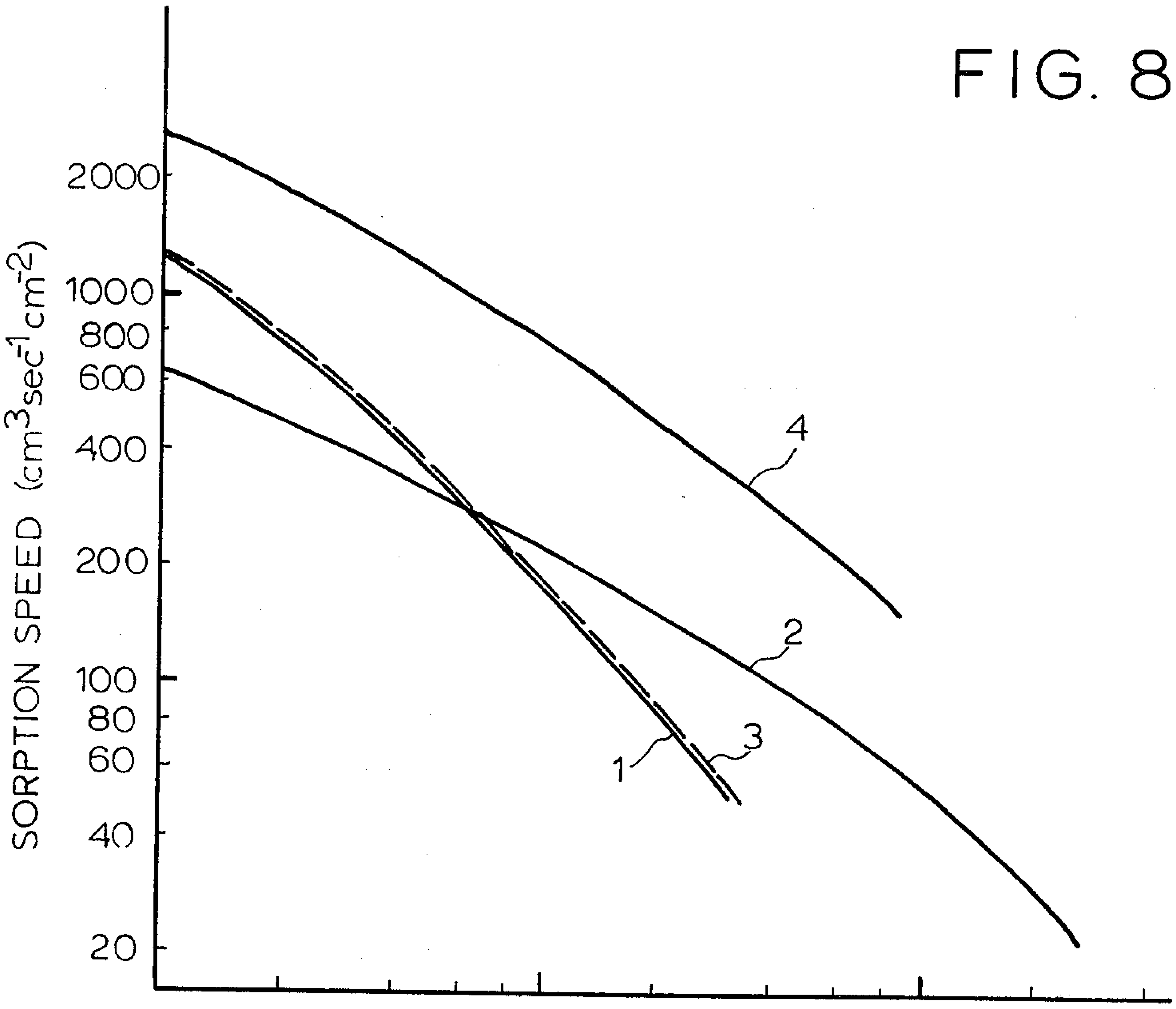

SORPTION SPEED (cm3sec-1cm-2)
2000
1000
800
600
400
200
100
80
60
40
20
4
2
1
3

FIG. 9

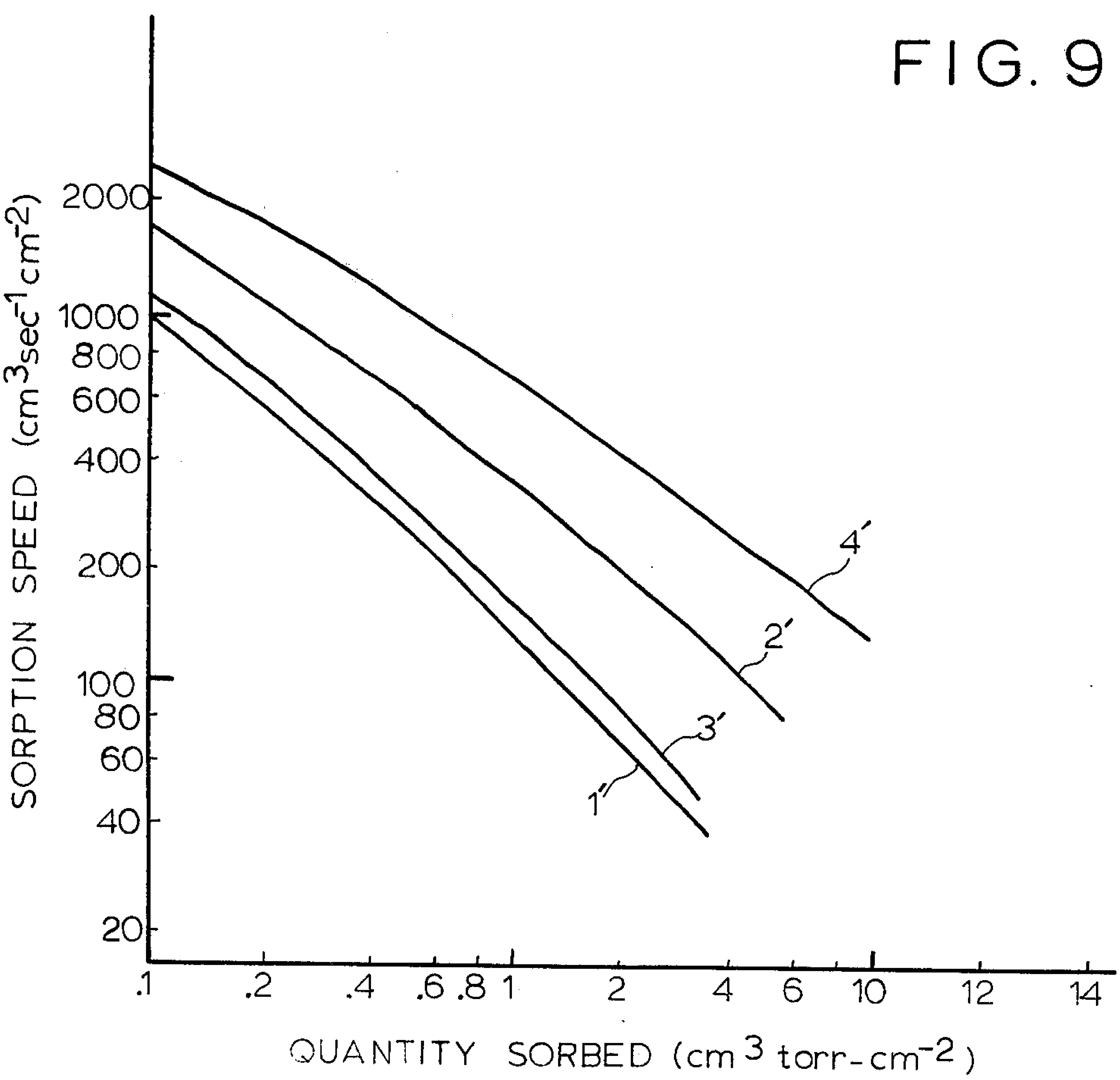

SORPTION SPEED (cm3sec-1cm-2)
2000
1000
800
600
400
200
100
80
60
40
20
.1
.2
.4
.6
.8
1
2
4
6
10
12
14
QUANTITY SORBED (cm 3 torr-cm-2)
4'
2'
3'
1'

FIG. 10

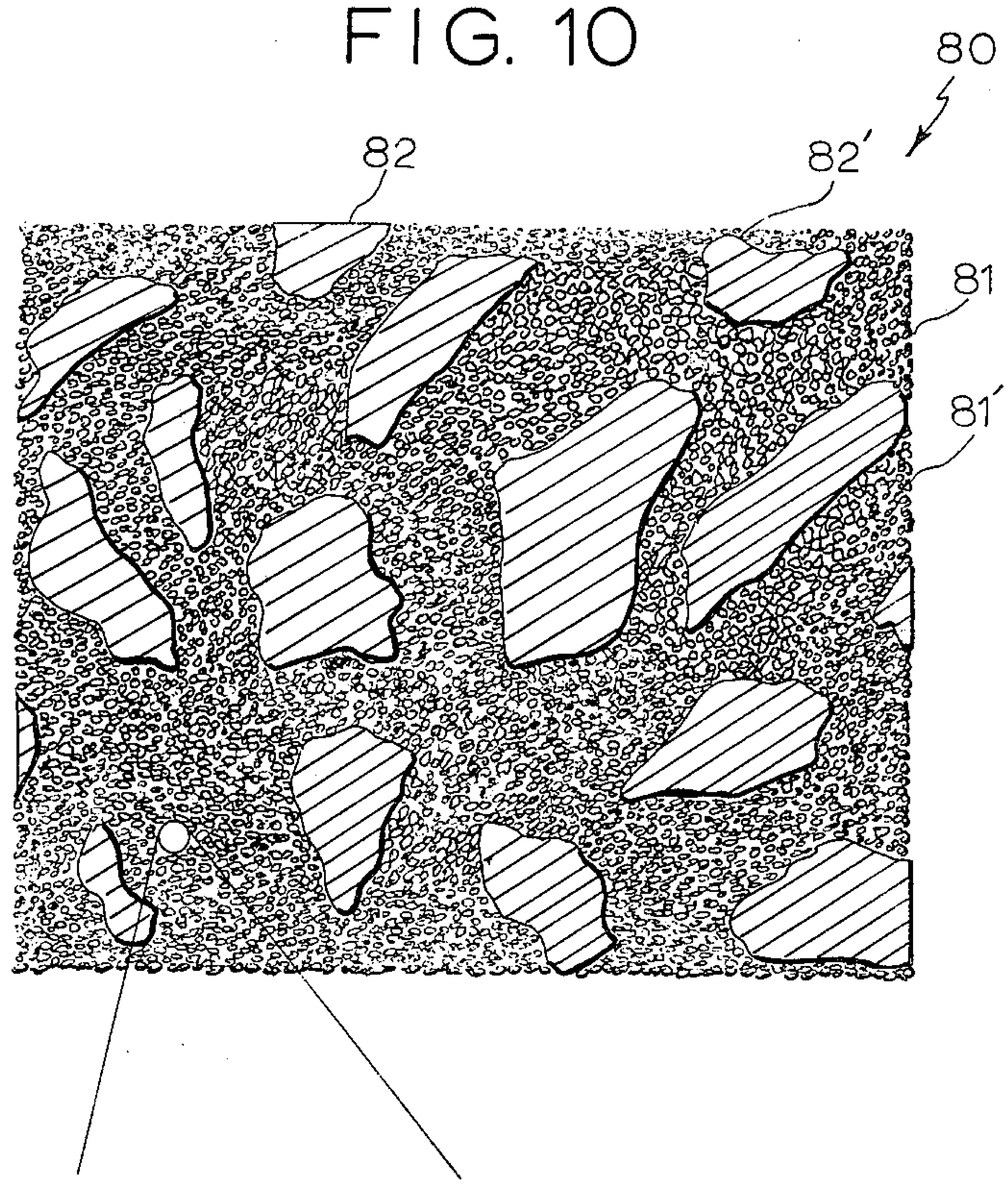
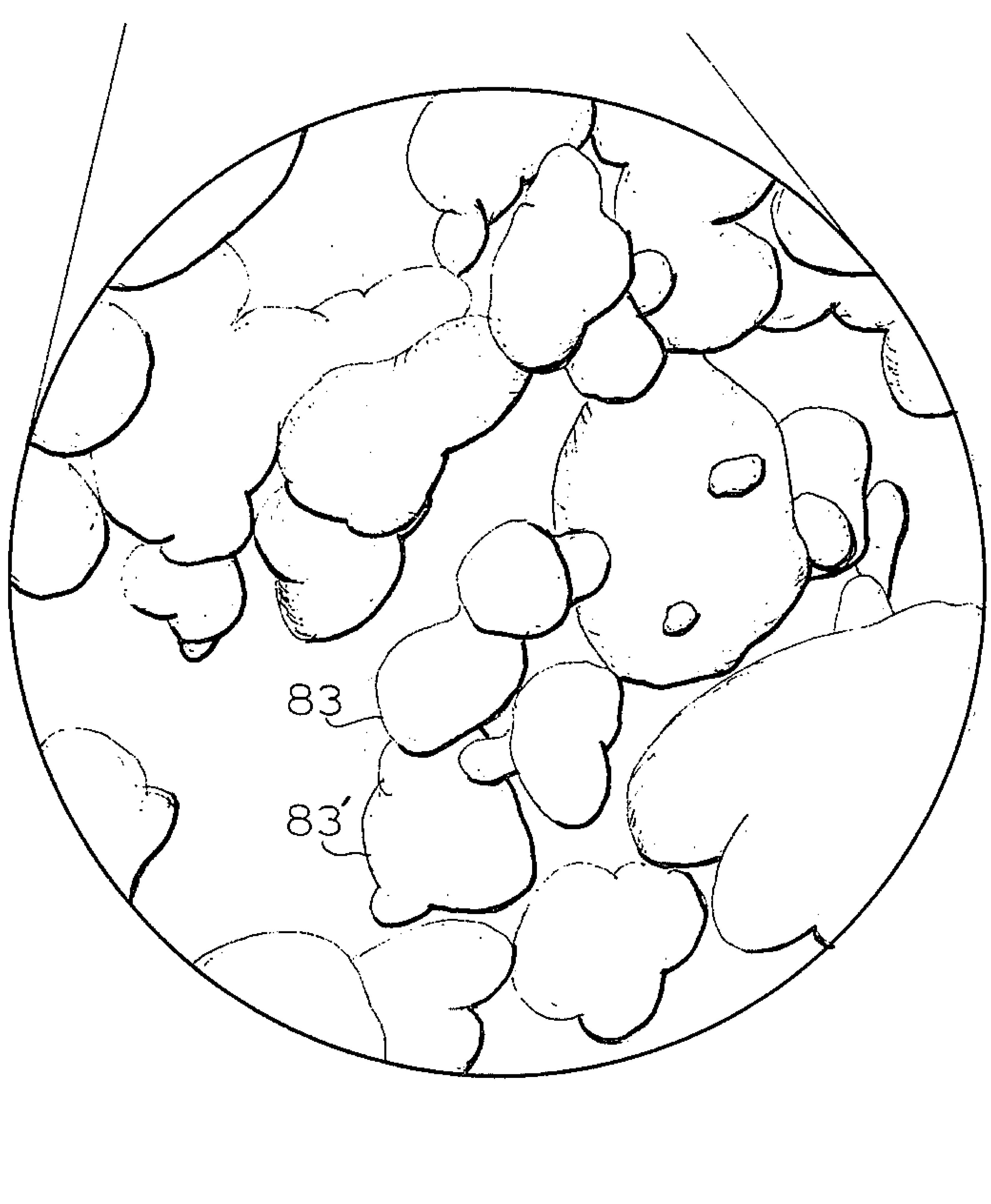


FIG. 11



GETTERING STRUCTURE

BACKGROUND OF THE INVENTION

The invention is directed to a getter device and composition for electrical discharge, vacuum and rare gas filled vessels employing a non-evaporable getter metal which preferably contains at least one metal selected from a group consisting of Zr, Ta, Hf, Nb, Ti, Th and U which is optionally heatable during operation of the vessel. In the past, such getter devices were constructed in the form of an open metal cup or pot-shaped vessel which was associated with an insulated heating coil of the type of an indirectly heated cathode with such metal vessel consisting of the getter metal or at least provided with a surface coating of such metal.

Getter devices employing zirconium, particularly correspondingly thick layers thereof produced by pressing and sintering of zirconium powder provide a considerably increased gas sorption speed and gas sorption capacity at temperatures above 600°C, but at intermediate and lower temperatures the gas sorption capacity is considerably limited by the fact that the gas diffusion into the interior of the zirconium is reduced whereby the gettering action is mainly due to the slight surface sorption of the zirconium. However, an increase of the gas sorption capacity of the getter at room temperature is absolutely necessary to ensure maintenance of the necessary vacuum or rare gas atmosphere of electronic tubes and other vessels under storage conditions.

An increase of the gas sorption capacity at room temperature can be achieved with a porous unpressed zirconium body, and in an effort to achieve greater porosity in sintered bodies of zirconium powder for getter purposes, molybdenum or tungsten powder was admixed with the zirconium powder. This arrangement, however, has the disadvantage, among others, that zirconium and molybdenum form an alloy at 1500°C (2732°F), as a result of which the sintering and degasification temperatures of such operating electrodes is considerably limited at the upper end.

Perdijk et al. in U.S. Pat. No. 2,855,368 suggest the addition of various powdered materials which react in a chemical or physical way with the powdered zirconium such as to reduce the temperature at which the activation of the zirconium takes place, thus reducing the probability of complete sintering. Among these additions are suggested aluminum, silicon, beryllium, tungsten, cerium and lanthanum. However, such reactions are not well controlled and a product of uncertain characteristics is obtained. In fact, in the same reference it is suggested to add a refractory metal powder such as tungsten to reduce the sintering of zirconium. $TiAl_3$ is also suggested as an antisintering agent.

Non-metallic antisintering agents have also been suggested such as by Wooten in U.S. Pat. No. 2,368,060 who adds powdered silica. In a further attempt to overcome the problems of sintering of the Zr powder non-evaporating getters Wintzer in U.S. Pat. No. 3,584,253 proposes the use of powdered graphite as an antisintering agent to maintain the large surface area of the active gas sorbing material. It shall be understood that these so-called "antisintering agents" do not prevent sintering, which is desirable in the present invention, but only retard sintering to a degree to make it more readily controllable.

Even though the introduction of poisonous gases into the electronic tube or other device by graphite is much reduced when compared with a similar gas transfer by the previously suggested metallic additions of molybdenum or tungsten powder, it must be realized that graphite can still introduce undesirable gases into the tube. Other antisintering agents such as refractory metallic oxides or other oxides such as silica are also known to introduce considerable amounts of poisonous gases into electronic tubes.

Furthermore, the additional antisintering materials proposed perform no other function apart from that of mechanically distancing the getter particles in such a way that sintering is reduced to a minimum. They also uselessly occupy space artifically increasing the volume of the getter composition.

Accordingly, it is an object of the present invention to provide an improved getter device and composition which is substantially free from one or more disadvantages of the prior art.

A further object is to provide a getter composition having at least equal gettering properties compared with traditional getter composition at room temperature and improved gettering properties at higher temperatures.

A further object is to provide a getter device having a means for preventing excessive sintering of the particulate powdered getter metal, said means itself performing a gettering function.

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

FIG. **1** is a top view of a getter device of the present invention;

FIG. **2** is a sectional view taken along line **2—2** of FIG. **1**;

FIG. **3** is a top view of a modified getter device of the present invention, and

FIG. **4** is a sectional view taken along line **4—4** of FIG. **3**;

FIGS. **5** and **6** are further getter devices;

FIG. **7** is yet another modification of a getter device;

FIGS. **8** and **9** are log-log graphs showing the sorption properties of getters of the present invention and corresponding properties for prior art getter compositions;

FIG. **10** is a schematic representation of a sectional view of the getter composition of the present invention magnified approximately 110 times;

FIG. **11** is an enlarged sectional view of the indicated portion of FIG. **10**, the diameter of FIG. **11** being approximately 40 microns.

According to the present invention there is provided a getter device comprising a holder which contains or supports a non-evaporable getter metal wherein said getter metal preferably comprises at least one metal selected from the group consisting of Zr, Ta, Hf, Nb, Ti, Th and U in intimate mixture with a zirconium-aluminum alloy. These zirconium-aluminum alloys are themselves non-evaporable getter materials and are characterized by (1) a sorptive capacity for noxious gases such as oxygen, carbon monoxide and water vapor, and (2) a vapor pressure at 1000°C of less than 10^{-5} torr. The preferred zirconium-aluminum alloys comprise from 5 to 30 and preferably 13 to 18 weight percent aluminum, balance zirconium. The most preferred Zr alloy is one of 16 percent aluminum, balance zirconium, available from SAES Getters S.p.A., Milan, Italy, under the trademark St 101. The present invention proceeds upon the concept of utilizing the Zr-Al alloy in conjunction with the non-evaporable getter metal in which the complete sintering of the particles of the getter metal is avoided during the heat treatment by employing Zr-Al alloy particles which are for example utilized by mixing Zr-Al alloy powder with the first getter material powder and applying the mixture to a support means. By the addition of Zr-Al alloy granules, for example, pressed layers with a higher porosity can be achieved than with ductile molybdenum or tungsten, and at the same time the poisonous gas transfer is lower than that of graphite. Getter devices of the present invention can be utilized with particular advantage in applications where a definite lack of space exists. In accordance with one method of the invention for producing getter devices of the type described, a heating means, already provided with a sinteredon insulation layer, is suitably coated with a mixture of powdered getter metal and powdered 16% weight Al-Zr alloy and subsequently heat treated in high vacuum at 800°–1200°C. The powder mixture may be in the form of an alcoholic suspension and applied by a dipping operation or the dry powder mixture may be pressed within a pressing die, at low pressure, and the molded material subsequently subjected to the desired heat treatment. Alternatively, the mixed getter powder may be supported as a layer of particles on at least one side of a supporting metal strip by a process as described by della Porta et al. in U.S. Pat. No. 3,652,317 or U.S. patent application Ser. No. 249,772 filed May 3, 1972. The powder may also pressed directly into a ring shaped holder well known in the art or may be painted, in the form of a liquid suspension, directly onto a suitable surface such as an electron tube electrode.

The getter devices and compositions produced according to the teachings of the present invention show a higher gas sorption speed and capacity at superambient temperatures compared to traditional getter devices and compositions using a graphite antisintering agent, in spite of the fact that the preferred Zr-Al alloy of 16% weight Al, balance zirconium, alloy is well known to sinter at the heat treatment temperatures of the present invention. This sintering is well described by della Porta in U.S. Pat. No. 3,203,901. The non-evaporable getter materials are characterized by (1) a sorptive capacity for noxious gases such as oxygen, carbon monoxide, and water vapor, (2) a vapor pressure at 1000°C of less than 10^{-5} torr. Examples of suitable non-evaporable getter materials include among others Zr, Ta, Hf, Nb, Ti, Th and U, mixtures thereof, alloys thereof with one another and with other metals, which alloys have satisfactory gettering properties. The preferred non-evaporable getter material is zirconium.

The non-evaporable getter metal and the Zr-Al alloy are preferably employed as finely divided particulate solids in intimate mixture one with the other. The weight ratio of the non-evaporable getter metal to the Zr-Al alloy is generally between 19:1 and 2:3 and preferably between 10:1 and 1:1. At higher ratios the Zr-Al alloy is not present in sufficient quantity to prevent excessive sintering of the non-evaporable getter metal. At lower ratios the getter metal is not present in sufficient quantity to perform its desired gas sorbing function especially at low temperatures. Both the Zr-Al alloy and the non-evaporable getter metal can be employed as particles of widely varying sizes. However, the non-evaporable getter metal is generally employed in particle sizes which pass through a screen of 100 mesh per inch and preferably those which pass through a screen of 200 mesh per inch. The Zr-Al alloy is generally employed in particle sizes which pass through a screen of 32 mesh per inch and preferably those which pass through a screen of 60 mesh per inch, and are retained on a screen of 100 mesh per inch.

The holder can be in any physical shape which will carry the getter composition. In one embodiment, the holder is an annular ring similar to the one commonly employed to hold vaporable getter metals such as barium. In another embodiment, the holder is a substrate which is preferably metallic and which has the particulate composition embedded in at least one of its surfaces.

The same substrate may be used as a support for other materials which might be useful within the tube such as mercury releasing materials.

In a further embodiment, the holder is in the form of a wire or rod around which is formed a pill or pellet of the getter composition.

The present invention is applicable to a wide variety of electron tubes, examples of which include, among other, radio receiving and transmitting tubes, X-ray tubes, television and radar kinescopes, klystrons, travelling wave tubes, mercury discharge tubes including fluorescent lamps. It is also applicable in rare gas purifiers, hydrogen purifiers, and in vacuum pumps.

Referring now to the drawings and in particular to FIGS. **1** and **2**, there is shown a device **10** of the present invention. In the getter device **10** the holder is in the form of an annular ring **11** having a cavity **12**, and a non-evaporable getter composition **13** within the cavity **12**.

Referring to FIGS. **3** and **4**, there is shown a getter device **30** which is connected to a similar getter device **30'** which in turn is connected to yet another similar getter device **30''**. The getter devices **30, 30', 30''**, etc., form a continuous running length of devices. In the device **30**, the holder is in the form of a substrate **31** having the getter composition **32** in particulate form partially embedded in the upper and lower planar surfaces of the substrate **31**. In operation, the getter device **30'**, for example, is separated from the devices **30** and **30''** by severing the substrate **31** in the vicinity of the small bridging attachments **33, 34, 35,** and **36.**

FIG. **5** shows a getter device **50** in the form of a pellet in which the holder is in the form of a rod **51** having the getter composition **52** compressed around and supported by said rod.

FIG. **6** shows a non-evaporating getter device **60** in the form of a pellet in which the holder **61** is an insulated wire of high ohmic resistance in the form of a heating coil **62** around which is formed the getter composition **63.**

FIG. **7** shows a non-evaporating getter device **70** wherein the holder consists of a wire spiral heatable by an electrical current and covered with an electrically insulating coating **72.** A covering of getter material **73** is applied by a method already described or by other methods well known in the art.

Referring now to FIG. **10**, there is shown a getter composition **80** of the present invention. The composition **80** comprises particles **81, 81'** of a sintered particulate non-evaporable getter metal. The composition also comprises particles **82, 82'** of a zirconium-aluminum alloy. As can be seen by reference to FIG. **10**, the particles **82, 82'** of the Zr-Al alloy are larger than the particles **81, 81'** of the getter metal. It can also be seen than the particles **82, 82'** of the Zr-Al alloy are distributed throughout the particles of the getter metals **81, 81'**. Furthermore, the particles of the Zr-Al alloy **82, 82'** are generally spaced out of contact with one another.

Referring now to FIG. **11**, there is shown an enlarged view of a portion of the particles **81, 81'** of FIG. **10.** As shown in FIG. **11,** particles **83, 83'** corresponding to particles **81, 81'** are in contact with one another and are sintered to one another. In accordance with the present invention, the particles **83, 83'** have a surface area after sintering which is substantially equal to and generally is at least 95% their surface area before sintering. Surface area measurements are made by the B.E.T. technique. See Volume LX of *The Journal of the American Chemical Society*, Feb. 1938, p. 309–319. See also Methods for the Determination of Specific Surface of Powders, Part I Nitrogen Adsorption (B.E.T. method) British Standards Institution (BS 4359:Part 1:1969). On the other hand, the sintering is conducted long enough in order to provide the composition with a compressive strength of at least 50 and preferably at least 300 kg/cm^2.

The invention is further illustrated by the following examples in which all parts and percentages are by weight unless otherwise indicated. These non-limiting examples are illustrative of certain embodiments designed to teach those skilled in the art how to practice the invention and to represent the best mode contemplated for carrying out the invention.

EXAMPLE 1

The tests of this example were performed to show the behavior of a prior art getter device. Particulate zirconium was mixed with particulate graphite as taught by Wintzer in U.S. Pat. No. 3,584,253 and then made into a fairly fluid paste in the form of an alcoholic suspension. A quantity of paste containing 100 mg of the powder mixture was placed in a ring holder to form a getter device **10** as illustrated in FIGS. **1** and **2.**

The getter device **10** was then placed in a vacuum of about 10^{-5} to 10^{-6} torr. The temperature was increased from room temperature to between 900° and 1100°C during a period of 25 minutes. The temperature between 900° and 1100°C was maintained for a further 5 minutes. The treated getter device was allowed to cool to room temperature and then removed from the vacuum furnace.

The getter ring **11** was attached to a thermocouple support and then mounted in a vacuum system, of design well known in the art and capable of reaching pressures less than 10^{-8} torr, to measure the gettering characteristics of the device. The whole system was then degassed by overnight heating of 350°C. When the pressure in the system was of the order of 10^{-8} torr the getter device was activated by heating the ring **11,** by means of high frequency heating, to 900°C for 10 minutes. When the system was again at a pressure of the order of 10^{-8} torr and the getter ring **11** had cooled down to room temperature, carbon monoxide was allowed to flow into the system through a conductance, C, of value 40cc/sec (for CO) in such a way that the CO gas pressure above the getter device, P*g*, was maintained at a constant value of 3×10^{-6} torr. At various intervals of time (t) the CO gas pressure (P*m*) at the conductance inlet, required to maintain P*g* at the constant value, was measured.

From the values of C, P*m*, P*g* and *t* obtained a curve of CO gas sorption rate as a function of total gas quantity sorbed by the getter material can be constructed. These results are shown in graphical form in FIG. **8** as curve **1.**

EXAMPLE 2

The procedure of Example 1 was followed in all respects except that during the CO sorption step the getter ring was maintained at 400°C by means of high frequency heating.

The results are shown in FIG. **8** as curve **2.**

EXAMPLE 3

The tests of this example were performed to show the behavior of getter devices of the present invention.

The procedure of Example 1 was followed in all respects except that the graphite was replaced by an equal volume of a zirconium-aluminum alloy of composition 16 weight percent aluminum, balance zirconium. In this example, the weight ratio of Zr to Zr-Al alloy is 3:2.

The results are shown in FIG. **8** as curve **3.**

EXAMPLE 4

The tests of this example were performed to show the behavior of a getter device of the present invention.

The procedure of Example 3 was followed in all respects except that during the CO sorption step the getter ring was maintained at 400°C by means of high frequency heating.

The results are shown in FIG. **8** as curve **4.**

DISCUSSION

It is seen from FIG. **8,** by comparing curve **2** with curve **4** and by comparing curve **1** with curve **3,** that at 400°C the getter materials of the present invention have a higher gas sorption speed, at a given quantity of gas already sorbed, than that of traditional getters and that at room temperature the gettering properties are at least equal.

The results shown in FIG. **8** have been confirmed by further experimentation since the filing of Italian application Ser. No. 28,053 A/72 on Aug. 10, 1972, now Italian Pat. No. 963,874, under which priority rights are claimed. The average results of the further experimentation are shown in FIG. **9** wherein curve **1'** corresponds to curve **1** of FIG. **8,** etc.

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 gettering structure comprising:

A. a sintered particulate non-evaporable getter metal selected from the group consisting of Zr, Ta, Hf, Nb, Ti, Th and U,

B. a particulate zirconium-aluminum alloy comprising 5 to 30 weight percent aluminum balance zirconium wherein the particles of the zirconium-aluminum alloy are larger than the particles of the non-evaporable getter metal and are distributed throughout the non-evaporable getter metal wherein the weight ration A:B is from 19:1 to 2:3 and wherein said particles of zirconium-aluminum alloy are generally spaced out of contact with each other.

2. A gettering structure comprising:

A. a sintered particulate non-evaporable getter metal, selected from the group consisting of Zr, Ta, Hf, Nb, Ti, Th and U,

B. a particulate zirconium-aluminum alloy comprising 5 to 30 weight percent aluminum balance zirconium wherein the particles of zirconium-aluminum alloy are larger than the particles of the non-evaporable getter metal and are distributed throughout the non-evaporable getter metal, wherein the sintered non-evaporable getter metal has a surface area after sintering substantially equal to its surface area prior to sintering wherein the weight ratio A:B is from 19:1 to 2:3 and wherein said particles of zirconium-aluminum alloy are generally spaced out of contact with each other.

3. A gettering structure comprising:

A. particulate zirconium, the particles of which pass through a U.S. standard screen of 200 mesh per inch,

B. a particulate alloy of 84 weight percent zirconium and 16 weight percent aluminum, the particles of which pass through a U.S. standard screen of 60 mesh per inch and are retained on a U.S. standard screen of 100 mesh per inch

with the provisos that:

1. the composition is sintered,
2. the composition exhibits a compressive strength of at least 300 kg/cm^2,
3. the total surface area of the zirconium particles after sintering is equal to at least 95% of their surface area before sintering;
4. the particles of zirconium are in contact with one another,
5. the particles of the alloy are distributed evenly throughout the particles of zirconium,
6. the particles of the alloy are generally spaced out of contact with one another,
7. the weight ratio of A:B is 10:1 to 1:1.

4. A gettering structure comprising:

A. sintered particulate zirconium,

B. A particulate zirconium-aluminum alloy comprising **5** to **30** weight percent aluminum balance zirconium wherein the particles of the zirconium-aluminum alloy are larger than the particles of zirconium and are distributed throughout the particles of zirconium in an amount such that the particles of zirconium-aluminum alloy are generally spaced out of contact with each other.

5. A gettering structure comprising:

A. sintered particulate zirconium,

B. a particulate zirconium-aluminum alloy comprising 5 to 30 weight percent aluminum, balance zirconium wherein the particles of the zirconium-aluminum alloy are larger than the particles of zirconium and are distributed throughout the particles of zirconium wherein the weight ratio A:B is from 19:1 to 2:3 and wherein said particles of zirconium-aluminum alloy are generally spaced out of contact with each other.

* * * * *

UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 3,926,832

DATED : December 16, 1975

INVENTOR(S) : Aldo Barosi

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

Col 3 line 17, delete "sinteredon" and insert -- sintered-on --.

Col 3 line 31, insert -- be -- between "also" and "pressed".

Col 4 line 23, delete "other" and insert -- others --.

Signed and Sealed this

sixteenth **Day of** *March 1976*

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks

REEXAMINATION CERTIFICATE (283rd)

United States Patent [19]

[11] **B1 3,926,832**

[45] Certificate Issued **Dec. 18, 1984**

Barosi

[54] **GETTERING STRUCTURE**

[75] Inventor: **Aldo Barosi**, Milan, Italy

[73] Assignee: **S.A.E.S. Getters S.p.A.**, Milan, Italy

Reexamination Request:
No. 90/000,450, Sep. 28, 1983

Reexamination Certificate for:

Patent No.:	**3,926,832**
Issued:	**Dec. 16, 1975**
Appl. No.:	**383,677**
Filed:	**Jul. 30, 1973**

Certificate of Correction issued Mar. 16, 1976.

[30] **Foreign Application Priority Data**

Aug. 10, 1972 [IT] Italy 28053/72

[51] **Int. Cl.**³ **H01J 7/18**; C22C 1/08; H01J 35/20; H01J 1/50

[52] **U.S. Cl.** .... **252/181.6**; 75/0.5 R; 75/246; 378/123

[58] **Field of Search** 252/181.6

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Primary Examiner—Ben R. Padgett
Assistant Examiner—Matthew A. Thexton

[57] **ABSTRACT**

A non-evaporating getter device and composition and process for producing such which is optionally heatable employing a getter material comprising at least one non-evaporable getter metal preferably selected from the group consisting of Zr, Ta, Hf, Nb, Ti, Th and U in intimate mixture with a zirconium-aluminum alloy.

REEXAMINATION CERTIFICATE ISSUED UNDER 35 U.S.C. 307.

THE PATENT IS HEREBY AMENDED AS INDICATED BELOW.

Matter enclosed in heavy brackets [ ] appeared in the patent, but has been deleted and is no longer a part of the patent; matter printed in italics indicates additions made to the patent.

AS A RESULT OF REEXAMINATION, IT HAS BEEN DETERMINED THAT:

The patentability of claims **1-5** is confirmed.

New claim **6** is added and determined to be patentable.

6. A gettering structure comprising:

A. sintered particulate zirconium;

B. a particulate zirconium-aluminum alloy comprising 5 to 30 weight percent aluminum, balance zirconium wherein the particles of zirconium-aluminum alloy are larger than the particles of zirconium and are distributed throughout the particles of zirconium such that the particles of zirconium-aluminum alloy are generally spaced out of contact with each other; and wherein the weight ratio A:B is from 19:1 to 2:3; and wherein the zirconium-aluminum alloy prevents excessive sintering of the zirconium, and wherein the total surface area of the zirconium particles after sintering is equal to at least 95% of their total surface area before sintering.

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