United States Patent

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[54] **METHOD AND VALVE APPARATUS FOR METERING THE FLOW OF LIQUID METAL**
10 Claims, 4 Drawing Figs.

[52] **U.S. Cl.** **137/1,** 251/144, 251/368, 266/38
[51] **Int. Cl.** **F27d 3/14,** F16k 5/04
[50] **Field of Search** .... 137/1, 572, 251, 246.23; 251/144, 309, 368; 266/(Consulted), 15, 16, 38, 39; 117/107; 118/(Consulted), 48, 49; 277/(Consulted), 135

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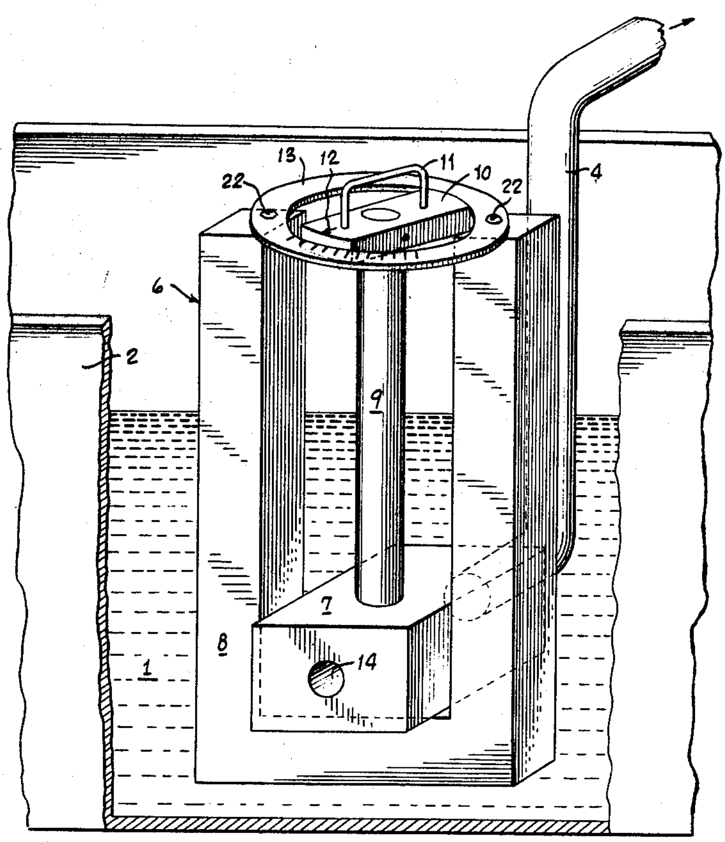
ABSTRACT: A method and means for metering the flow of liquid zinc between a reservoir and a vacuum chamber in a vapor deposition process, comprising a valve body and operator structure composed of graphite which is immersed in the liquid zinc in the reservoir so that the body of zinc acts as an atmosphere-to-vacuum seal, and as a result of the nonwetting of graphite by zinc at the operating temperatures of the deposition process a liquid-to-liquid seal is also achieved within the valve structure.



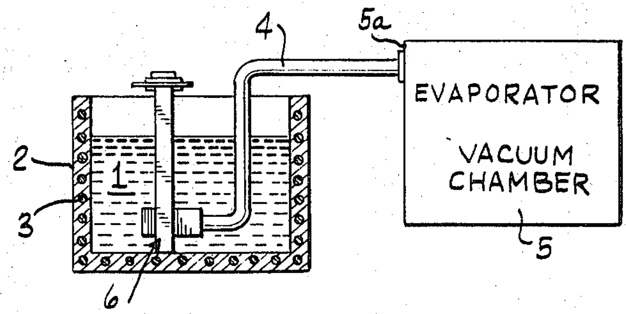


Fig. 1.

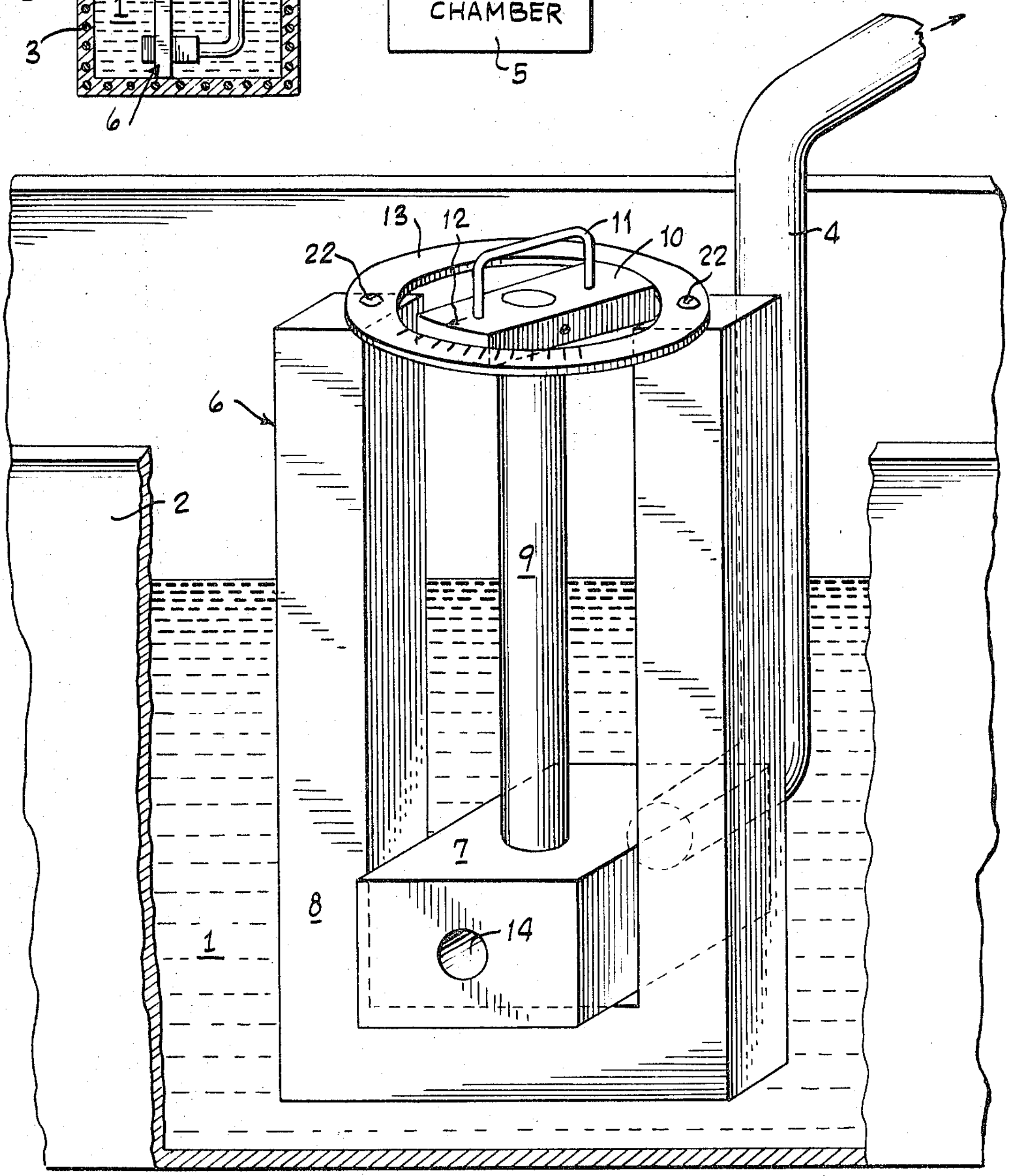


Fig. 2.

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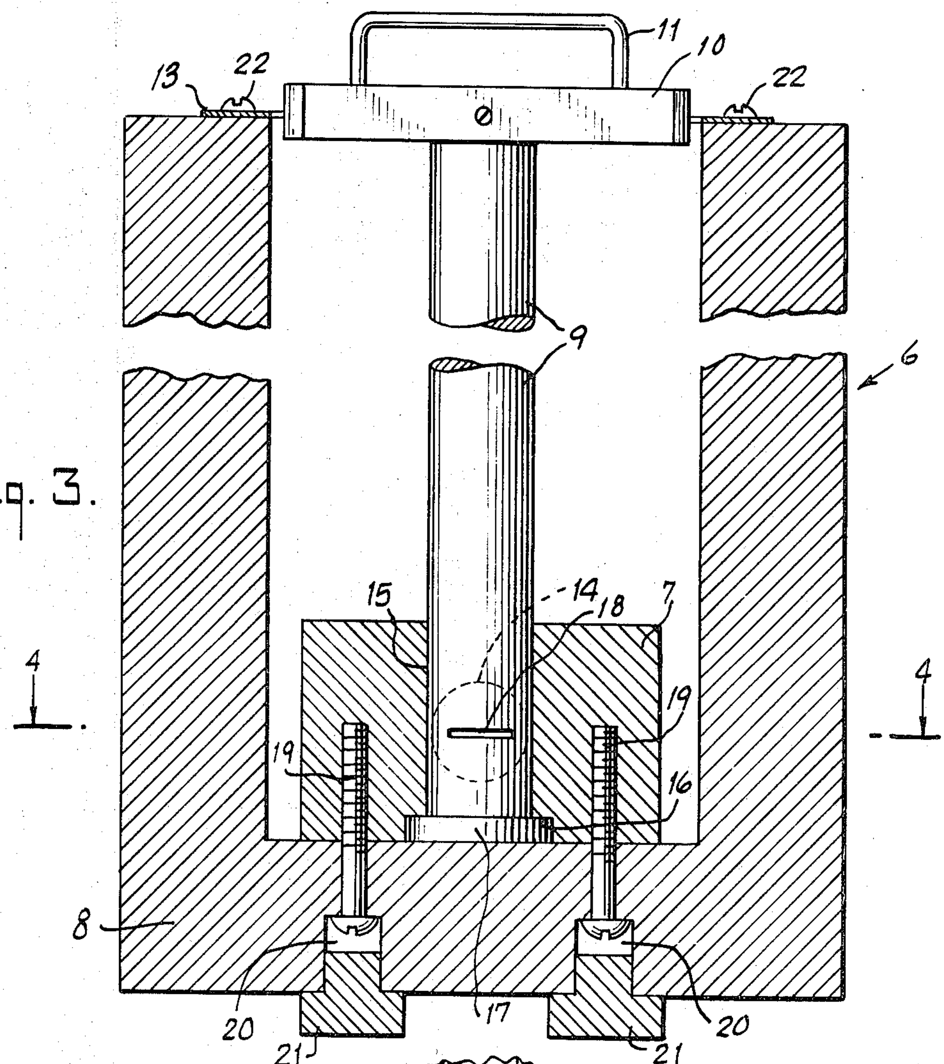

Fig. 3.

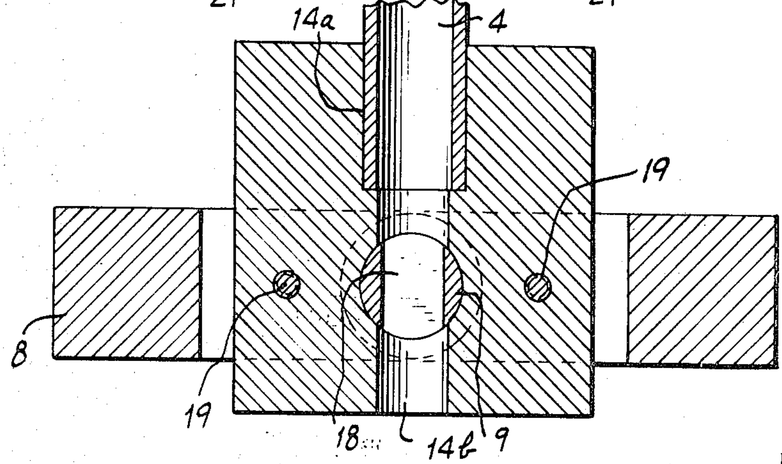

Fig. 4.

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METHOD AND VALVE APPARATUS FOR METERING THE FLOW OF LIQUID METAL

BACKGROUND OF THE INVENTION

The present invention relates to the fluid flow control art and more particularly to a valve means for controlling the flow of liquid metal to a vacuum chamber in a vapor deposition process.

It has long been a problem in the materials handling field to control the flow of highly corrosive and chemically active materials. The valving to be used in such environments requires special packing and sealing as well as the use of special materials of construction. Such valves are generally rather expensive due to their complicated construction and the use of special materials in their manufacture, and have a comparatively short working life.

A particularly difficult valving application in this art is controlling the flow of a liquid metal to be used as a coating in a vapor deposition process. In this process, a selected substrate which is to be coated is placed in a heated vacuum chamber and the metal for the coating is introduced in liquid form at an inlet. The liquid metal upon exposure to the vacuum evaporates by virtue of the decreased pressure and increased temperature and subsequently is deposited by condensation on the substrate forming a thin even layer thereon. The process is commonly used for example in coating zinc on steel. The valve controlling the flow of the liquid zinc in the process must necessarily resist the highly corrosive effects of this molten metal and operate at the attendant high temperatures. Moreover, such a valve must provide sealing against the great difference in pressure between the ambient atmosphere and the vacuum in the chamber in which the deposition takes place. If air is permitted to enter the chamber, the zinc tends to oxidize and form undesirable deposits within the chamber environment.

To the best of my knowledge, no appropriate valves are commercially available for use with the process as none has been found which is sufficiently vacuum tight.

The valve of the present invention effectively meters the flow of liquid metals, such as zinc, between a reservoir and a vapor deposition chamber, while maintained at a temperature above the melting point of the metal, and yet remains vacuum tight and leakproof although of comparatively simple and inexpensive construction. In addition, it is of sufficient durability to be comparatively maintenance-free and resists corrosion by or amalgamation with the liquid metal being conducted.

SUMMARY OF THE INVENTION

The valve of the invention is constructed of a refractory material, such as graphite, and comprises a valve body, a support frame, and a valve stem or operator having a control handle. The entire valve structure, with the exception of the handle, is immersed in the liquid metal which acts to seal the joints against leakage of air into the subatmospheric pressure region at the outflow end of the valve. Also, by virtue of the nonwetting quality of particular liquid metals with respect to refractory materials, such as graphite, at the required operating temperatures, there will be no liquid leakage through the various clearances between the valve components.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a showing of a vacuum deposition system in which the valve of the present invention is used;

FIG. 2 is a perspective vie of the improved valve shown in its operating position in a liquid metal reservoir with the parts of the reservoir broken away for clarity;

FIG. 3 is a sectional view of the improved valve means; and

FIG. 4 is an enlarged view in section showing the means for metering the flow of the metal.

DETAILED DESCRIPTION

The valve of the present invention will be described in connection with a system for vapor depositing a layer of zinc on a steel substrate, but it will be understood that a number of similar metals, which are capable of use in combination in the vapor deposition process may selected as is known to those skilled in the art.

FIG. 1 shows a vapor deposition system of the type in which the improved valve means is to be used. The supply of zinc **1**, which is to be deposited, is maintained in the liquid state in a reservoir **2**. The reservoir **2** may be a large open container such as a commercially available melting pot containing resistive wire **3** as the heating means. The zinc is maintained at a temperature between 788° F., its melting point, and up to about 1100° to 1200° F., at which point it begins to oxidize in the atmosphere. The molten zinc **1** is delivered through a pipe **4** to a heated, evaporator or vacuum chamber **5**. The pressure in the chamber is maintained in the range between 10^{-3} and 10^{-4} millimeters of mercury. The steel substrate on which the zinc is to be deposited is located in this chamber **5**. The liquid zinc upon entering the vacuum chamber **5** evaporates, by virtue of the decreased pressure and increased temperature, and is subsequently deposited by condensation on the substrate forming a thin, even layer thereon. The flow of zinc **1** between the reservoir **2** and the vacuum chamber **5** is controlled by a valve means **6** which is the subject of the present invention.

The valve means **6** is shown in more particular detail in FIG. 2 positioned in the reservoir **2**. The valve means **6** comprises a valve body **7** and a stem or operator **9**, both mounted on a U-shaped frame **8**. The stem **9** extends from the valve body **7** and has a crossbar member **10** fastened to its upper end. The crossbar member **10** has a handle **11** mounted on its upper surface by which the stem **9** may be operatively rotated. An arrow **12** is marked at one end of the crossbar **10** and cooperates with an indicator dial **13** mounted on the upper ends of the arms of the frame **8**. The indicator dial **13** is calibrated to give an indication of the amount of liquid zinc being metered by the valve **6** for any given amount of stem rotation.

The valve outlet accepts a delivery pipe **4** which conducts the zinc to the chamber **5**. The pipe **4**, which is constructed of chromium carbide treated steel to resist the action of the zinc, extends upwardly through the surface of the zinc **1** and over the edge of the reservoir **2** for connection to the vacuum chamber **5**. Sealing of the entrance port **5***a* in the vacuum chamber **5** is achieved by welding the supply pipe **4** in the wall of the chamber **5**, or by other suitable vacuum connections.

The valve body **7**, the stem **9** and the frame **8** are all constructed of a refractory material, preferably graphite. It has been found that under the operating conditions of the deposition process the graphite is not wetted by the liquid zinc. As a result of this phenomenon the valve may be submerged in the liquid zinc without danger of corrosion or other damage at operating temperatures of 788° F. and above, and no zinc will enter the clearances between the cooperating parts. With the valve submerged in the body of zinc, the zinc acts to seal out the air and to seal itself against leakage within the valve thus serving as a structural seal element. No further sealing is necessary so the construction of the valve parts may be simple and uncomplicated, requiring no special structure, materials, or casting.

Accordingly, as shown in greater detail in FIGS. 3 and 4, the valve body **7** may be in the form of a block with a bore **14** through its center. The delivery pipe **4** is pressfitted in an enlarged end **14***a* of the bore **14**, as shown in FIG. 4, and a tight seal results from the thermal expansion of the pipe **4** when subjected to the temperature of the liquid zinc **1**.

Another bore **15** is provided in the valve body **7** perpendicular to the central bore **14** for accommodating the cylindrical stem or operator **9** which extends through the central bore **14**, as best seen in FIG. 3. A cutout portion !6 **16** in the lower part of the bore **15** receives an annular flange **17** on the lower end of the stem **9** to prevent longitudinal motion of the stem **9** with respect to the body **7** while permitting rotational motion therein. The frame **8** supports the stem **9** within the valve body **7**. The stem **9** is provided with an opening **18** extending transversely therethrough which cooperates with the central bore **14** in the valve body **7** to meter the flow of zinc from inlet **14***b* to the tube **4** as will be presently described. It has been found that if the valve tolerances are within generally acceptable limits, such as 0.004 inch or less, no leakage will occur in the valve system.

The valve body **7** is held on the frame member **8** by two screws, **19**, one at either side of the central bore **14**. The underside of the frame is appropriately bored at **20** to accept the mounting screws **19** and plug members **21** are fitted in these bores **20** to prevent the liquid zinc from acting directly on the material of the screws. These plug members **21** also act as footings for the frame **8**, spacing it from the floor of the reservoir **2**. The upper arms of the frame **8** are bored to accept screws **22** which hold the indicator dial **13** thereon, and the upper end of the stem **9** is bored to receive a screw securing the handle **10** thereto.

The metering operation of the valve of the present invention is as follows. The valve body **7** is totally immersed in the liquid zinc **1** so that the clearances at the entrance and exit openings of the central bore **14** and the operator bore **15** are all bathed by the zinc **1**. The supply pipe **4** is fitted in the exit opening **14***a* before insertion of the valve means **6** into the zinc. The stem **9** is oriented with the axis of its transverse opening **18** perpendicular to the axis of the central bore **14** so as to block or shutoff flow through the valve body **7**.

When it is desired to supply zinc to the evaporator **5**, the stem **9** is rotated by means of the handle **11** thus bringing opening **18** into registration with bore **14**. The amount of zinc which is permitted to pass into the tube **4** will be a function of the extent of rotation of the stem **9**. The stem **9** is free to rotate in either direction to increase or decrease the degree of registration between bore **14** and stem opening **18** thereby metering the amount of flow. A person operating the valve will have an indication of the amount of flow passing through the valve for a given setting by observing the point on the indicator dial **13** coinciding with the marking **12** on the crossbar member **10**. Flow rates in a range from 10 to 75 pounds per minute have been achieved with such a device but almost any flow rate can be obtained depending on the valve dimensions.

It will be seen that the system is sealed against the influx of air into the valve means **6** and the delivery pipe **4** by means of the zinc itself which acts to seal all of the valve clearances. The opposite end of the delivery pipe **4** is sealed by welding to the chamber wall so that air cannot enter the evaporator at this point so that undesirable oxidation and deposits within the deposition system due to air leakage are avoided. Since the valve body **7**, the stem **9** and the frame **8** are all constructed of graphite, as already explained, all of the openings in and clearances between these respective members are graphite to graphite contact surfaces. In this particular system, the zinc is maintained at a temperature of approximately 850° F., but is has been found that at all the working temperatures used with this process, zinc will not wet the graphite. Therefore, the zinc itself will act to seal the various clearances between the parts also preventing any fluid leakage.

My invention contemplates the use of other materials in constructing the valve, such as silicon carbide, zirconia, alumina, Stellite 06, Molybdenum-30 percent Tungsten alloy, and others, but graphite is particularly suitable since it does not require expensive casting and may be readily machined. Graphite will resist a large variety of liquid metals, acids, and other corrosive fluids and can withstand temperatures in excess of the evaporation temperatures of any metal which might be metered by the valve.

Thus, an improved method and means is provided, comprising a valve of simple and inexpensive construction, which selectively meters the flow of a liquid metal to a vacuum deposition chamber and which seals itself and the chamber in a superior manner without requiring complicated construction or special materials.

We claim:

1. A system for metering the flow of a liquid metal from a reservoir comprising:
   a. a supply of the liquid metal to be metered in the reservoir;
   b. a valve body of refractory material positioned in the reservoir below the level of the metal and having a passage therein for accommodating the flow of the metal between an inlet and an outlet below the level of the metal;
   c. an operator of refractory material in said passage for metering the flow of metal of said outlet;
   d. means external to the metal for controlling the movement of said operator in said passage;
   e. means for maintaining the temperature of the metal within a range wherein the metal does not wet the refractory material and thereby does not enter and seals the clearances between the valve body and the operator; and
   f. conduit means connected to said outlet for conducting the metered metal out of said reservoir.

2. A system as claimed in claim 1 wherein the refractory material is graphite.

3. A system as claimed in claim 1 wherein said conduit means is of a chromium carbide treated steel.

4. A system as claimed in claim 1 wherein said operator comprises a member having an opening therein, said member being movable in said passage to meter the flow of said metal in proportion to the degree of registration of said opening and said passage.

5. A system for metering the flow of liquid zinc between a reservoir and a chamber at a pressure below atmospheric comprising:
   a. a supply of the liquid zinc to be metered in the reservoir;
   b. a valve body of graphite positioned in the reservoir below the level of the zinc and having a first passage therein for accommodating the flow of the zinc between an inlet and an outlet in said body and a second passage intersecting said first passage between said inlet and said outlet;
   c. an operator of graphite positioned in said second passage for metering the flow of metal to said outlet, the clearances between said operator and said valve body being no greater than .004 inch;
   d. means external to said zinc for controlling the movement of said operator in said second passage;
   e. heating means for maintaining the temperature of the zinc approximately within the range from 788° F. to 1200° F. such that the zinc will not wet the graphite, sealing the clearances between the valve body and the operator; and
   g. a tube fitted in said outlet and extending out of said zinc for connection to said chamber to conduct the zinc from said reservoir to said chamber.

6. A system as claim in claim 5 wherein said tube is of chromium carbide treated steel.

7. A system as claimed in claim 5 wherein said operator comprises a cylindrical column having an opening therein, said column being movable in said second passage to meter the flow of the zinc to the outlet in proportion to the degree of registration of said opening and said first passage.

8. A system for metering the flow of a liquid from a reservoir, comprising:
   a. a valve body adapted to be positioned in the reservoir below the level of the liquid to be metered and having a passage therein for accommodating the flow of the liquid between an inlet and an outlet;
   b. an operator in said passage for metering the flow of the liquid to said outlet;
   c. means external of said liquid for controlling the movement of said operator in said passage;
   d. the valve body and operator material being such that they are not wetted by the liquid so that no liquid enters and thus the liquid seals the clearances between the valve body and the operator; and

e. conduit means connected to said outlet and extending out of said reservoir for conducting the liquid from said reservoir.

9. The method of metering and sealing the flow of a liquid from a reservoir to an environment below atmospheric pressure comprising the steps of:

a. providing a valve having an inlet and an outlet and of a material which is not wetted by the liquid to be metered;

b. connecting one end of a conduit to the outlet of said valve;

c. immersing the valve in the reservoir with all the clearances and the outlet below the level of the liquid and with the other end of the conduit extending out of the liquid; and

d. connecting the external end of the conduit to the environment below atmospheric pressure.

10. The method as claimed in claim 9 wherein the valve material is graphite.