Nov. 2, 1954 P. ALEXANDER 2,693,521

HEATER FOR VACUUM METALIZING APPARATUS

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FIG. 1

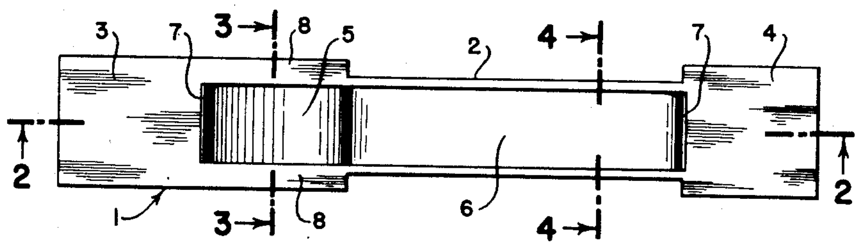

FIG. 2

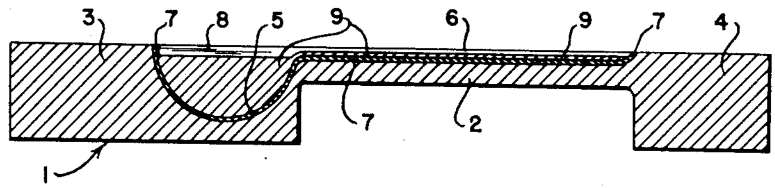

FIG. 6

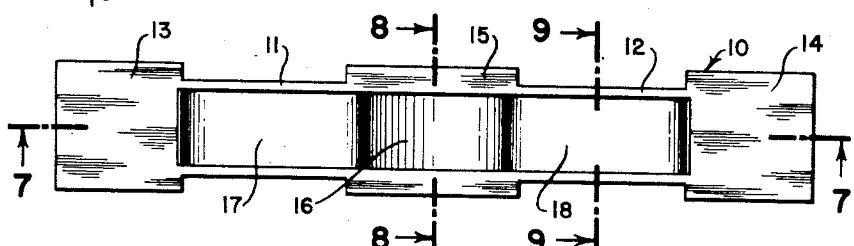

FIG. 7

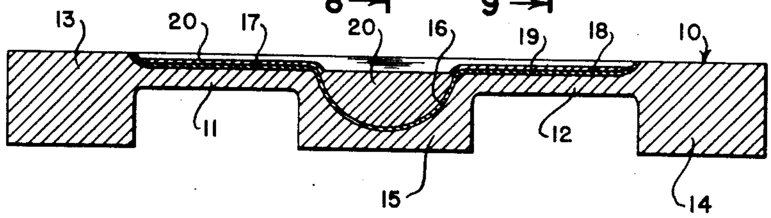

FIG. 3

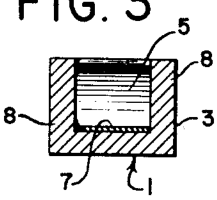

FIG. 4

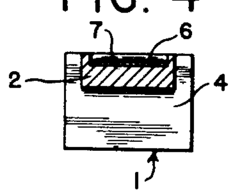

FIG. 5

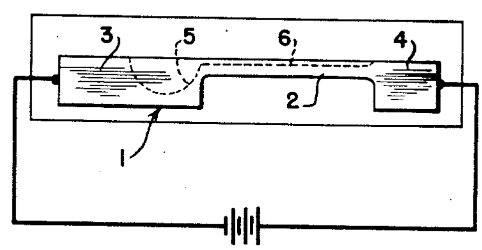

FIG. 8

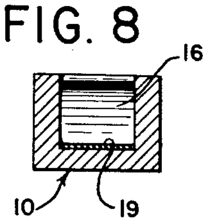

FIG. 9

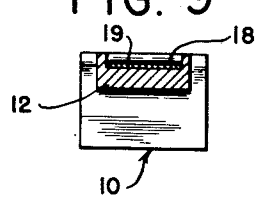

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FIG. 10

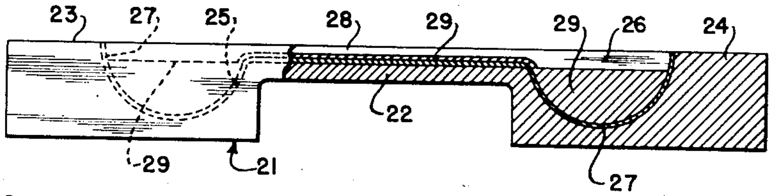


FIG. 11

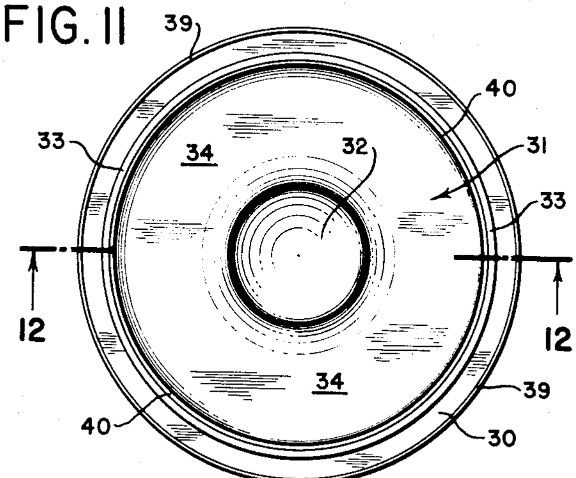
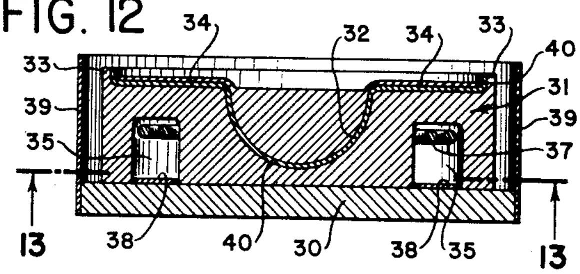


FIG. 12



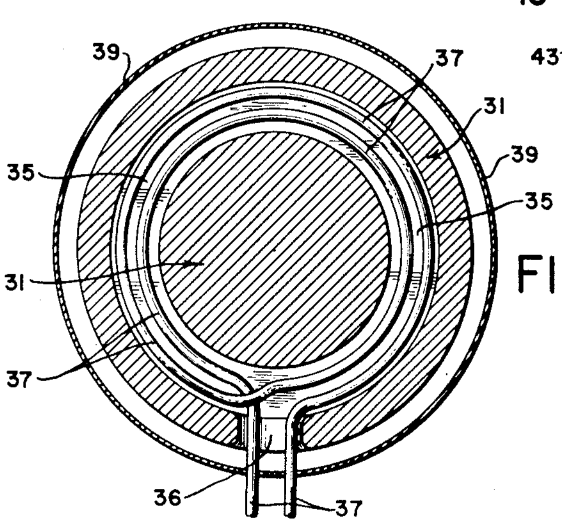
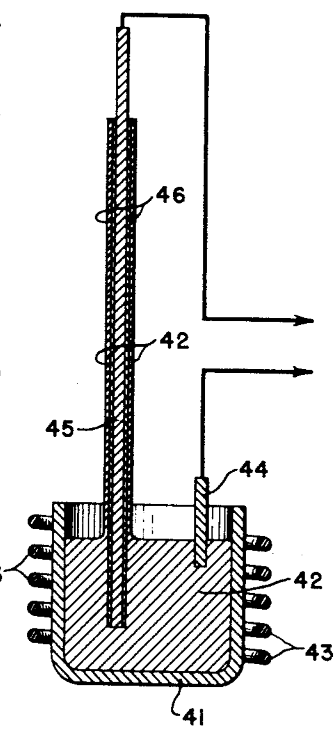


FIG. 13

FIG. 14



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HEATER FOR VACUUM METALIZING APPARATUS

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Application December 26, 1951, Serial No. 263,297

3 Claims. (Cl. 201—63)

This invention relates to apparatus for vaporizing metal in vacuum, and the general object is to provide novel and highly efficient means for vaporizing metal in connection with apparatus of this class.

The act of applying a film of metal to various surfaces by condensing metal vapor upon such surfaces has been the subject of considerable research, and much success has been achieved. However, development of heater elements for evaporating the metal to be deposited has not heretofore kept pace with that of other elements of apparatus for applying metallic film by condensation of metallic vapor.

United States Patents Nos. 2,153,786, granted April 11, 1939, and 2,444,763, granted July 6, 1948, cover methods and apparatus for vacuum evaporating aluminum wire which is fed onto a heater element. In these patents the metal wire is fed against an incadescent tungsten filament, in vacuum, whereby the leading end of the advancing wire is heated to a high temperature and so vaporized. This method and apparatus has enjoyed commercial success in the art of depositing metallic film by condensation of metallic vapor, on various surfaces. However, only that portion of the incandescent filament in contact with the advancing wire performs useful work, and energy utilized to maintain other portions of the filament at high temperature is a loss. Further, those portions of the filament not in contact with the wire are of much higher temperature than that portion employed in evaporating metal of the wire, and in consequence, a highly undesirable amount of excess heat is present at the heating element. Shielding the radiant heat of the non-working portions of the filament is not practical. The excessive heat tends to liberate vapors and gases from the exposed portions of apparatus for depositing metallic film or from material upon which the metallic vapor is to be condensed; and the presence of such liberated vapors and gases interferes with obtaining and maintaining the low degree of pressure essential for satisfactory operation of the apparatus. Further, in the presence of such excessive heat, certain materials desired to be coated, such as paper and plastic film, are apt to be damaged. A further objection to the use of the tungsten filament is that such filament is eroded by some metals being vaporized.

Objections inherent in the use of incandescent tungsten filaments were largely overcome by the improved heater element disclosed in application for United States Patent bearing Serial Number 208,162, filed January 27, 1951. Herein, instead of tungsten filament, the heater element comprises a carbon rod in series with a suitable circuit, the rod being coated with one of two mixtures which render the heater surface wettable by molten metal whereby the metal rapidly spreads from the point of contact to cover the entire coated surface of the heater element. Thus evaporation takes place all along the rod, and no excessively hot, non-working areas exist.

Evaporating elements of the crucible type comprising a container heated directly or indirectly to the temperature needed for the evaporation of the metal have the disadvantage that unless the container is very large the rate of evaporation of the metal must be comparatively slow. The rate of evaporation of the metal in a high vacuum depends on the area of the exposed surface of molten metal, and the temperature of the metal. The area of the exposed surface of the metal is limited by the size of the crucible, and if the temperature of the metal is too high the metal has the tendency to bubble and to be ejected

out of the crucible. This is especially the case in metals of low specific gravity, like aluminum.

A particular object of the present invention is to provide an improved heater element for apparatus of the class described, said element having two functionally distinct parts, one part providing a crucible or container for the metal to be evaporated and the other part providing a surface or surfaces wettable by the metal, and accessible to the metal, and from which the metal is evaporated. It is advantageous to maintain the evaporating surface or surfaces at a higher temperature than that of the metal within the container, for it has been found that this facilitates the creeping of the molten metal from the container to said surface or surfaces. In fact the container part of the element need not be maintained at a temperature much higher than the melting point of the metal.

Thus, the crucible or container part of the element has practically the sole function of holding the metal to be evaporated in a liquid state, and this part of the element may be kept at a temperature sufficient only to melt the metal. This eliminates the disadvantages of evaporating directly from a crucible, since, in the present heater element high temperature and large area of the crucible are both unnecessary.

The function of the other part of the element, that is, the wettable surface or surfaces accessible to metal creeping thereon from the crucible or container, is to provide for the exposure of a large area of molten metal whereby evaporation may take place at a high rate; and such surface or surfaces are preferably maintained at a much higher temperature than the crucible.

The molten metal charge of the crucible progressively creeps out and onto the evaporating surface or surfaces until the charge is exhausted. The rate of operation of the heater element is a function of the area of the evaporating surface or surfaces, and of the related temperature.

An important feature of the invention is that the reservoir or crucible may be adapted to hold enough metal to permit a relatively long run of the apparatus before exhaustion of the charge. Further the crucible may be repeatedly or continuously replenished by simple means within a vacuum. For example, granular metal may be, from time to time, dumped into the crucible before an earlier charge has been exhausted; or a wire may be continuously fed into the crucible at a rate corresponding with that of evaporation.

A further object is to provide a heater element comprising a crucible and an evaporation surface having long life, so that apparatus of the class described may work uninterruptedly for a considerable period of time.

There are two general methods by which this invention may be carried out.

One is by using an evaporating unit consisting of the two described parts which is a conductor of electric current and can be heated to the desired temperature by passing current through it, and the other is by using an element which is heated indirectly, for example, as by a resistance heating element distinct from the evaporating element itself, or by induction heating, electron bombardment, or any other known method suitable for heating in vacuum, or combinations of such means.

In the first case, in which the element is heated by electric current passing through it, the element is preferably of an elongated shape and the functionally different portions of the element form resistances in series through which the current passes. This element has to be designed in such a way that the crucible part has a substantially lower resistance than the evaporating surface so that this surface will be heated to a higher temperature than the crucible. This is facilitated by the molten metal contained in the crucible which has a low electrical resistance. Such an element will function satisfactorily even if the resistance of the crucible is negligible compared to the resistance of the evaporating part, and the current heats essentially only the evaporating part because the metal in the container can be kept in the molten state by heat conducted to it from the evaporating part only.

In the second case in which the element is heated indirectly it is only a matter of design to make it sure that the evaporating part is heated to a higher temperature than the crucible part.

The described elements can be made of a number of electrical conductors but it is desirable that such conductor should have a comparatively high electrical resistance, comparatively low heat conductivity and that it should be suitable for the construction of the required shape. Metals are not advantageous because due to their high heat conductivity it is difficult to maintain a large enough temperature differential between the crucible and evaporating surface. Carbon is a particularly suitable material because of its comparatively high electrical resistance and its suitability for the forming of suitable shapes. In order to facilitate wetting of the carbon by the metal and in some cases to prevent chemical reaction between the carbon and the metal, it is desired to provide the carbon surfaces with a coating which is wettable by the metal and if necessary which protects the carbon from the metal. Such a coating is, e. g., a combination of titanium carbide and metallic titanium, or zirconium carbide and metallic zirconium.

Other high refractory semi-conductors like metallic carbides and nitrides are also suitable materials for the evaporating elements. Examples are the carbides and nitrides of titanium, zirconium, hafnium, vanadium, tantalum, and columbium.

These, and other objects and features of the invention, will be more fully understood from the following description and the drawing.

In the drawing:

Fig. 1 is a plan view of one form of the invention, while Figs. 2, 3, and 4 are respectively the sections **2—2**, **3—3**, and **4—4** of Fig. 1;

Fig. 5 is a plan view of another form of the invention, Figs. 6 and 7 being respectively the sections **6—6** and **7—7** of Fig. 5;

Fig. 8 is a plan view of a third form of the invention, Figs. 9 and 10 being respectively a bottom plan view of this form and the section **10—10** of Fig. 8;

Fig. 11 is a plan view of a fourth form of the invention, Fig. 12 being the section **12—12** of Fig. 11;

Fig. 13 shows a fifth form;

Fig. 14 shows a sixth form.

A simplified form of the invention is illustrated in Figs. 1 through 4.

In this form a bar of electrically conductive material, preferably of relatively high resistance, such as carbon, is utilized as the base of the apparatus, and is adapted to be placed in series with a circuit of sufficiently high amperage.

In these figures, such a bar is designated **1**, and may be of carbon of rectangular cross section, as shown. The bar is of reduced cross section throughout an intermediate portion **2**, the two end portions **3** and **4**, respectively to the left and right in Figs. 1 and 2, being of greater cross section. It will be noted that end portion **3** is of greater length than end portion **4**.

A crucible **5** is provided by recessing the end portion **3** near the intermediate portion **2**; and a shallow channel **6** is provided in the intermediate portion **2**. As a convenience in manufacture the crucible **5** may be formed by milling the end portion **3** downwardly to the desired depth with a cylindrical cutter whose axis is normal to the longitudinal axis of the bar. The channel **6** may then be formed by the same either by first raising the same a desired amount and then moving the bar lengthwise against the cutter.

The cylindrical surface of the crucible and the bottom of the channel are, herein, coated with material **7** which is wettable by molten metal, the material being preferably a mixture of titanium carbide and metallic titanium.

A circuit having the apparatus in series therewith is shown diagrammatically in Fig. 5. The apparatus is, of course, a resistance furnace of particular construction and function. When the circuit is closed, the heat generated in the bar is relatively small at the end portions due to the large cross section of these portions; but in the greatly reduced intermediate portion the resistance is considerable, and, in the presence of sufficient amperage, this portion may be brought to a high temperature while the end portions of the bar remain at a relatively low temperature.

The presence of the crucible in end portion **3** of the bar diminishes somewhat the effective cross section of that portion; however, the side walls **8**, **8** of the crucible may be fairly thick, as shown; and the finished bar may readily be designed so that, in operation, the crucible temperature may be near the melting point of the metal to be vaporized while the intermediate portion **2** is at or above the boiling point of the metal—the determining factor being, of course, merely cross sectional differences. In operation the crucible, heated in some measure by its own resistance to the passage of current, is also further heated by conduction from the extremely hot intermediate portion **2**. This conduction heating is of importance because, obviously, when the crucible is charged with metal, and the metal is molten, the resistance at the crucible may drop considerably due to the additional current path provided by the metal, and conduction heat from the intermediate portion **2** is required to maintain the crucible above the melting point of the metal.

In Fig. 2 the metal **9** to be vaporized is shown in molten condition. The metal may be introduced into the crucible in any suitable manner. For example a granular charge may be loaded into the crucible; or, with the crucible heated beyond the fusing point of the metal, a bar or wire of the same may be fed into the crucible so that the leading end of the bar or wire is melted therein to fill the same. The device may be adapted for continuous operation if means are provided for feeding a bar or wire of metal or metal in granular form into the crucible at a rate according with the evaporation rate.

Molten metal present upon a surface which is differentially heated will migrate or creep preferentially toward the area of highest temperature. It is, however, necessary that the molten metal wet the heated surface. If the metal wets the surface, the fused metal will even creep up hill to reach a zone of highest temperature. In order that a metal to be vaporized, such as aluminum, may have a wettable track upon which it may creep when molten, the coating of the combination of titanium carbide and metallic titanium is provided herein.

Molten aluminum, for example, will not "wet" a carbon surface except at excessive temperatures; but if the surface is coated with the above described mixture, an entire charge of metal within the crucible will be vaporized within time although the molten metal is obliged to creep up hill out of the crucible into the channel. Molten metal received within the channel spreads evenly; vaporization takes place only above the channel and not above the cooler crucible.

A modification of the invention is shown in Figs. 6 through 9.

In this modification a bar of electrically conductive material is also used. The bar, here designated **10**, may be of the same material as bar **1**. Bar **10** is preferably of rectangular cross section, the general cross-section being reduced throughout two like intermediate portions **11** and **12** so that end portions **13** and **14** and a central portion **15** are of greater and equal cross section. A crucible **16** is provided in central portion **15**, and extending away from the crucible **16** along intermediate portions **11** and **12** are, respectively, shallow channels **17** and **18**. The crucible **16** and the channels **17** and **18** may conveniently be formed in the bar by milling.

In operation bar **10** is inserted in series in a suitable circuit; and the hottest portions of the bar are plainly the two intermediate portions **11** and **12**; due in part to reduction of cross-section at the crucible, but principally due to conduction of heat from the adjacent intermediate portions **11** and **12**, the next hottest part of the bar will be that part forming the crucible **16**; and the terminals **13** and **14** will be relatively cool.

A coating **19** of a mixture of titanium carbide and metallic titanium is provided on the bottoms of channels **17** and **18** and on the cylindrical surface of crucible **16**.

The metal **20** to be vaporized is indicated as in molten state in Fig. 7.

In this structure it will be plain that the molten charge creeps in opposite directions from the crucible **16**, as shown in Fig. 2.

It is somewhat difficult to prepare a bar, such as **10**, so that the two intermediate portions **11** and **12** are of precisely the same cross section. If, for example, portion **11** is of less cross section than portion **12**, the hottest part of the bar will be, initially, portion **11**. The molten metal will tend to creep more rapidly from the crucible into channel **17** than into channel **18**. However, when channel **17** is filled before channel **18**, the presence of the molten metal in channel **17** will reduce the resistance of intermediate portion **11**, and portion **12**, then of least effective cross section, will become the hottest part of the bar. The preponderant creep direction will then reverse; the molten metal will favor channel **18**. This compensatory reversal of creep will rectify any slight irregularities of manufacture of the bar **10**.

Fig. 10 shows a modified form of heater element comprising a resistance bar **21** having an intermediate portion **22** of reduced cross section, and end portions **23** and **24**, both of which are recessed to provide crucibles **25** and **26** respectively. The coating **27** of the mixture of titanium carbide and metallic titanium extends from crucible **25** across a shallow channel **28** in portion **22** to crucible **24**. In this structure more storage capacity for metal to be vaporized is provided, the vaporization, of course, taking place on the bridging intermediate portion **22**. This portion, the hottest part of the bar, heats both crucibles by conduction. The metal is designated **29**.

Another form of the invention is shown in Figs. 11, 12, and 13.

Mounted on a suitable circular base **30** is discoid **31** in the center of the upper surface of which is a relatively deep depression constituting a crucible **32**. The remainder of the upper surface of the discoid is slightly depressed, except for a rim **33**, whereby an annular vaporizing surface **34** is provided. The under side of the discoid is provided with an annular depression **35** having a radial depression **36** of equal depth extending from depression **35** to the periphery of the discoid. The annular depression is registered with the annular vaporizing surface **34**. Within the depression **35** is a heating element **37**, the terminals of which are passed through depression **36**. Mounted on base **30** and registered with depression **35** is a metallic reflector **38**, which may be a flat annulus, as indicated in Fig. 12. Mounted on the outside of base **30** and surrounding the same is another reflector, **39**. The crucible **32** and the annular vaporizing surface **34** are coated with a mixture **40** which may also be of titanium carbide and metallic titanium.

Metal charged into the crucible **32** is therein melted and creeps radially up onto the annular surface **34** where it is vaporized. It will be seen from the sectional figure that the annular surface **34** is the hottest part of the discoid, although the heating element also operates to bring the crucible to high temperature. By proper design the apparatus may be adapted to heat the crucible sufficiently to melt the metal, and the annular surface **34** to vaporizing temperature.

Another form of the invention is illustrated in Fig. 14. Herein a crucible is designated **41**. The crucible, into which the metal **42** to be vaporized is first melted, is adapted to be heated by element **43**, here shown as a coil of resistance wire. A flow of current is maintained through the molten metal within the crucible by means of electrodes **44** and **45** immersed in the melt. Electrode **44** is merely for purposes of contact. Electrode **45** is a resistor, preferably an elongated carbon rod, coated with a mixture **46** of titanium carbide and metallic titanium. The heating element **43** is designed to maintain metal within the crucible in molten state. Electrode **45** is designed to create a temperature in excess of the boiling point of the metal. Here the molten metal within the crucible progressively creeps up on electrode **45** and is vaporized.

While only a few forms of the invention have been illustrated and described, it will be understood that a crucible, adapted to be heated in any suitable manner to somewhat above the melting point of the metal to be employed, may be variously combined and cooperate with an evaporation surface adapted to be heated in any suitable manner to the vaporizing point of the metal. Further, while only a wetting agent comprising titanium carbide mixed with metallic titanium has been specifically mentioned, this agent may be substituted for by any of a number of other agents—for example, a mixture of vanadium carbide and metallic vanadium, zirconium carbide and metallic zirconium, or even the carbide of the metal employed. Also the entire heater element may be made of material which is wettable by the metal to be evaporated.

I claim:

1. Apparatus for evaporating metal in vacuum which comprises a circuit having in series therewith an elongated conductor of greater resistance than said circuit, said conductor having end portions and a central portion of relatively large cross section and intermediate portions, one thereof being between one of said end portions and said central portion, and the other thereof between said central portion and the other of said end portions, said intermediate portions being of relatively small cross section, said central portion constituting a crucible and said intermediate portions constituting runways for molten metal flowing from said crucible; and a coating of material wettable by molten metal, said coating covering at least part of the inside of said crucible and extending therefrom along said runways.

2. Apparatus for evaporating metal in vacuum, which comprises a circuit having in series therewith an elongated carbon bar, said bar having end portions and a central portion of relatively large cross section and intermediate portions, one thereof between one of said end portions and said central portion, and the other thereof between said central portion and the other of said end portions, said intermediate portions being of relatively small cross section, said central portion constituting a crucible and said intermediate portions constituting runways for molten metal flowing from said crucible; and a coating of material wettable by molten metal, said coating covering at least part of the inside of said crucible and extending therefrom along said runways.

3. Apparatus according to claim 2 wherein said coating comprises a mixture of titanium carbide and metallic titanium.

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