

March 12, 1968

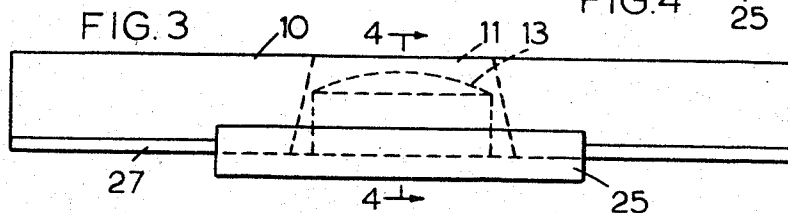
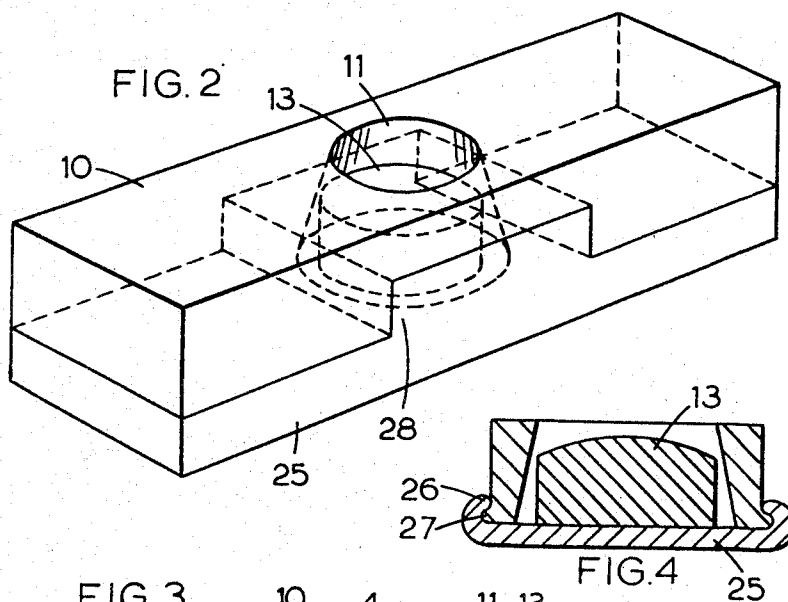
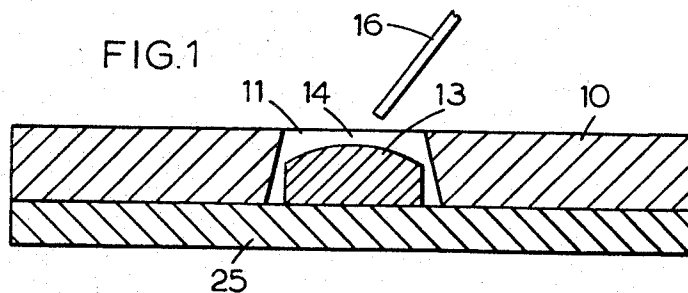
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3,373,260

VAPORIZATION OF METALS AND METALLOIDS

Filed April 26, 1965

2 Sheets-Sheet 1



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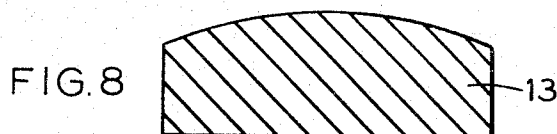
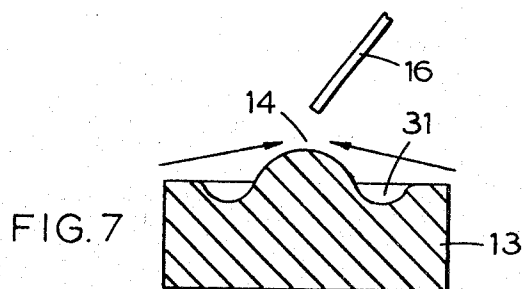
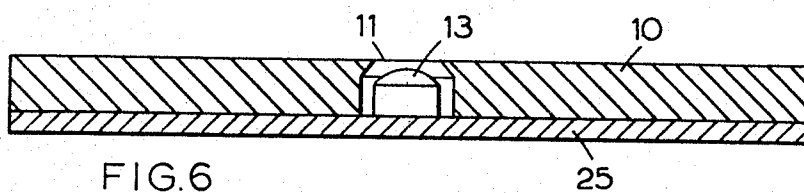
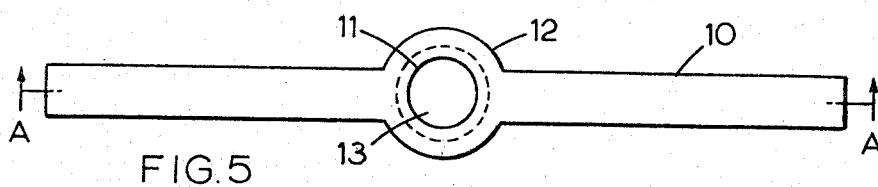
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3,373,260

VAPORIZATION OF METALS AND METALLOIDS

Filed April 26, 1965

2 Sheets-Sheet 2



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1

3,373,260

VAPORIZATION OF METALS AND METALLOIDS  
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Filed Apr. 26, 1965, Ser. No. 450,730

Claims priority, application Great Britain, Apr. 30, 1964,  
17,963/64

13 Claims. (Cl. 219—275)

## ABSTRACT OF THE DISCLOSURE

The disclosure provides an electrical heater which supports a plug for evaporating aluminium or other volatilisable material, the plug being contained in a recess in the heater which recess is smaller at the upper part than the lower part so that the plug will not rise out of the recess due to electromagnetic action.

This invention relates to the evaporation of metals or metalloids, e.g. aluminium, chromium, nickel and silicon. Such evaporation is required for the plating of articles by vapor deposition.

One method for effecting evaporation of metals and metalloids comprises the use of an electrically conducting bar or elongated crucible or boat called the "evaporation source," clamped between two electrodes, through which current is passed which heats the source by electrical resistance so that metal fed on the latter is evaporated, for example as described in British Patents Nos. 770,751 and 770,752.

Many metals and metalloids are highly corrosive at the temperatures of evaporation.

The characteristics best suited for the heating function, such as suitable electrical resistance, mechanical strength, and thermal and mechanical flexibility, are often not found in the constructional materials suited for the function of best chemical resistance to the substances which are to be evaporated.

It is, therefore, often advantageous to separate these two functions, for example as described in British Patent No. 770,753, by using an electrical resistance heating element, hereafter, referred to as "the heater" having a recess in which is inserted a removable refractory plug or similar insert, hereafter called "the plug."

The heater may be made from graphite or from refractory carbide, boride, nitride, oxide or a refractory metal like tungsten or molybdenum or mixtures thereof, or other refractory material. The plug may be made from a refractory carbide, boride, nitride, oxide (for example, tantalum carbide) or mixtures thereof, or other material resistant to attack by the material to be evaporated.

The "material" (which term hereafter is intended to include metals and metalloids) to be evaporated is fed to the centre of the top surface of the plug where it collects as a pool of molten metal and vaporization is effected by heat conducted to the metal pool from the surface of the heater. Solid metal is commonly fed in the form of a wire to the plug.

One reason for keeping the plug as a detachable insert, is that the metal to be evaporated usually attacks the article from which the material evaporates. Economies are, therefore, effected by using a multiplicity of plugs in the same heater or, in appropriate cases, the heaters can be made from a cheap substance such as graphite and a multiplicity of heaters may be used with the same plug.

In existing arrangements, the upper face of the plug is either flush with, or above the upper face of the heater (e.g. FIGURE 3 in British Patent No. 770,753).

I have found that attack by the material on the plug

2

is concentrated in areas where liquid material collects as a molten pool on the plug surface, but that attack by vapor of the material on the plug is either absent or small and generally less than attack by the liquid material. Liquid material may also attack the heater especially by contact with the surface of the heater in the recess.

According to the present invention, a heater suitable for evaporating volatilisable material has a recess to receive a plug from which the material can be evaporated, said recess being smaller at the top than at the bottom thereof.

This invention makes it possible to use a plug which tends to levitate due to an electromagnetic field produced by electrical heating of the heater. The operating temperature is such as is necessary to vaporize the material. For example, if the material is aluminium, a suitable temperature of said zone is in the region of 1650–1700° C. for which purpose the heater may be raised to a temperature of about 1900 to 2000° C.

In order to create this zone, it is necessary that the top of the plug shall be below the plane of the surface of the heater at the top edges of the recess. The distance between the top of the plug and the top of the recess may be varied within wide limits, e.g. 0.01 to 0.8 of the depth of the recess, or in most cases 0.1 to 0.3. The upper face of the plug may, for example, be flat or dome shaped.

Although the invention minimises contact of liquid material with the plug, such contact may accidentally occur, e.g. on starting up or due to irregular material feed or current fluctuation, and the present invention also provides special forms of plug which avoids molten material reaching the heater.

It is important to preserve the plug surface substantially intact, i.e. to prevent or minimise crater formation (namely, the opposite of the effect shown in FIGURE 1 of British Patent No. 770,753). If a crater is formed in a plug, the material to be evaporated is shielded from heat radiated by the heater, and receives then substantially only conducted heat which induces accumulation of molten material in the crater and a progressively increasing rate of corrosion.

The material may be fed continuously or intermittently towards a recess in an electrically heated heater which recess contains a plug, the feed and the temperature being such that the solid material is not contacted with the plug but is vaporized above the plug.

According to another aspect of the present invention, the recess in the heater is shaped to provide an abutment against which the plug can contact to prevent its being ejected from the recess by the magnetic field of the electric current used for heating. This aspect of the invention permits the use of lightweight compounds for the plug, for example titanium di-boride. Earlier methods excluded the use of lightweight plug materials although some of them, e.g. titanium di-boride, have very desirable resistance to the attack by metals at elevated temperatures, and they may also be cheaper.

If a lightweight plug is used, the plug is inserted into the heater orifice from below and is held in position by a suitable support, which support need not be a conductor of electricity. This triple arrangement, which separates the functions of the plug, the heater and the support, has the following added advantages.

The mechanical load is carried by the support which can, therefore, be selected in a material of best mechanical strength at lowest cost without the limitation of having to allow for suitable electrical characteristics.

The heater, which is carried by the support, can be selected substantially on the basis of most favorable electrical characteristics.

A particular advantage of dividing the heater in this way is that the resulting geometrical form becomes then much

3

more resistant to thermal stresses, namely improved thermal flexibility, which manifests itself in practice by a multiple operational life.

The arrangement will now be further described by way of example with reference to the accompanying diagrammatic drawings, wherein:

FIGURE 1 is a sectional view of a heater made in accordance with the invention together with the plug;

FIGURES 2 and 3 show modifications to be described;

FIGURE 4 is a section on line 4—4 on FIGURE 3;

FIGURE 5 is a plan view showing a modified form of heater;

FIGURE 6 is a section A—A on FIGURE 5, showing also the support; and

FIGURES 7 and 8 show modified shapes of plugs.

Referring first to FIGURE 1, a bar shaped heater 10 made of graphite is provided with a conical recess 11, the upper end of which is smaller than its bottom end. In the recess 11 is a plug 13 which is cylindrical in plan and has a height less than that of the recess so as to leave a high temperature zone 14 above the plug within the recess. A rod or wire 16, of material to be vaporized, is fed into the hot zone 14 and is thus vaporized by radiated heat. The function of the plug is relegated to that of the removable safety insert in the heater so that material, which accidentally may not have been vaporized, will fall on to the detachable plug insert instead of contacting the heater with consequent risk of attack on the heater.

The heater permits the use of mechanically light materials (for example titanium di-boride) for the plug inserts.

The plug 13 is inserted through the bottom of the heater orifice 11. If an electromagnetic field induces levitation of the plug, the plug is prevented from being ejected from the heater or orifice.

The heater is made in two parts, viz the heater part 10 having the recess 11 and a lower part 25 which may be made of stronger material (e.g. vitreous carbon, alumina, or the like) and serves to support the part 10 and also to support the plug 13. The parts 10 and 25 may be clamped together by any suitable means, or support 25 only may be clamped with the heater 10 sitting on support 25.

FIGURE 2 shows a modified arrangement also having a supporting member 25. This permits also versatility, with respect to the wall thickness of the heater 10 around the orifice 11 to produce highest electrical resistance in the desired areas, namely, immediately above the plug 13. The supporting member 25 has a projection 28 engaging a corresponding slot in the heater part 10 in the region of the recess. Recess 11 is partly in the heater and partly in the member 25.

The supporting member 25 which carries the plug 13, can also be held in position by other means. For example, FIGURE 3 shows a side view of such an arrangement and FIGURE 4 is a transverse sectional view of the arrangement of FIGURE 3. In this example the heater has at the bottom end, protruding ribs 27 over which upstanding flanges 26 at the edges of the supporting member 25 can be slid.

FIGURES 5 and 6 show an arrangement similar to that of FIGURE 1 but with the orifice 11 cylindrical in the lower part and frusto-conical only near the upper end. One object of this arrangement is to ensure that the distance between the plug 13 and the orifice 11 is either substantially uniform or slightly greater towards the top end, so as to minimise any tendency for vapors to be trapped near the lower end of the orifice 11.

A thin disc can, if required, be inserted below the plug. This disc can be made from a suitable material, e.g. vitreous carbon.

4

The plug, for the purpose of the present invention, may be cylindrical, cubic or of other shape.

Heating may be effected, if desired, by means other than electrical resistance heating, e.g. by high frequency heating.

As shown in FIGURE 5 the walls 12 may be thinner than the remainder of the bar 10 in plan view.

I claim:

1. An electrical resistance heater suitable for evaporating volatilisable material having a recess, a plug disposed in said recess, from which plug the material can be evaporated, said recess being smaller at the top than at the bottom thereof to prevent expulsion of the plug by levitation.
2. A heater as claimed in claim 1, wherein the recess is frusto-conical.
3. A heater as claimed in claim 1, wherein the recess is frusto-conical at the upper part thereof and cylindrical at the lower part thereof.
4. A heater as claimed in claim 1, wherein the plug is formed of a light-weight material such as titanium di-boride.
5. A heater as claimed in claim 1, having the recess extending completely therethrough so that the plug can be inserted from below and a separate supporting member is provided to support the plug in the recess, the heater being made of a material suitable for electrical resistance heating while the supporting member is made of a different material having better mechanical properties than the heater material.
6. A heater as claimed in claim 5, wherein the heater and the supporting member have inter-engaging ribs and flanges for sliding engagement.
7. A heater as claimed in claim 1, in combination with an evaporation source in the form of a plug in the recess of less height than the recess so that a zone for high temperature evaporation is provided in the recess above the plug so as to minimise or avoid formation of molten material on the plug.
8. The combination claimed in claim 7, wherein the distance between the top of the plug and the top of the recess is from 0.1 to 0.3 of the depth of the recess.
9. The combination claimed in claim 7, wherein the top of the plug is dome shaped.
10. The combination claimed in claim 7, wherein the plug has an annular depression in its upper end.
11. The combination claimed in claim 7, wherein the recess in the heater extends completely therethrough and the heater has projecting means at the lower part of the recess to support the plug.
12. A heater as claimed in claim 1, wherein the wall thickness of the heater at the recess is reduced at the lower part thereof.
13. A heater as claimed in claim 5, wherein the supporting member has a projecting part extending into the heater in the region of the recess, the recess being partly in the heater and partly in the supporting member.

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