

March 19, 1957

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2,785,997

GAS PLATING PROCESS

Filed March 18, 1954

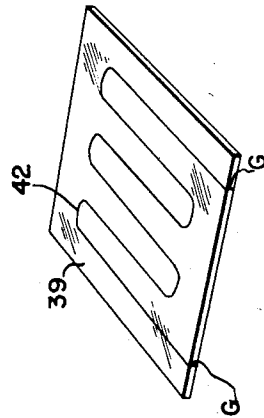
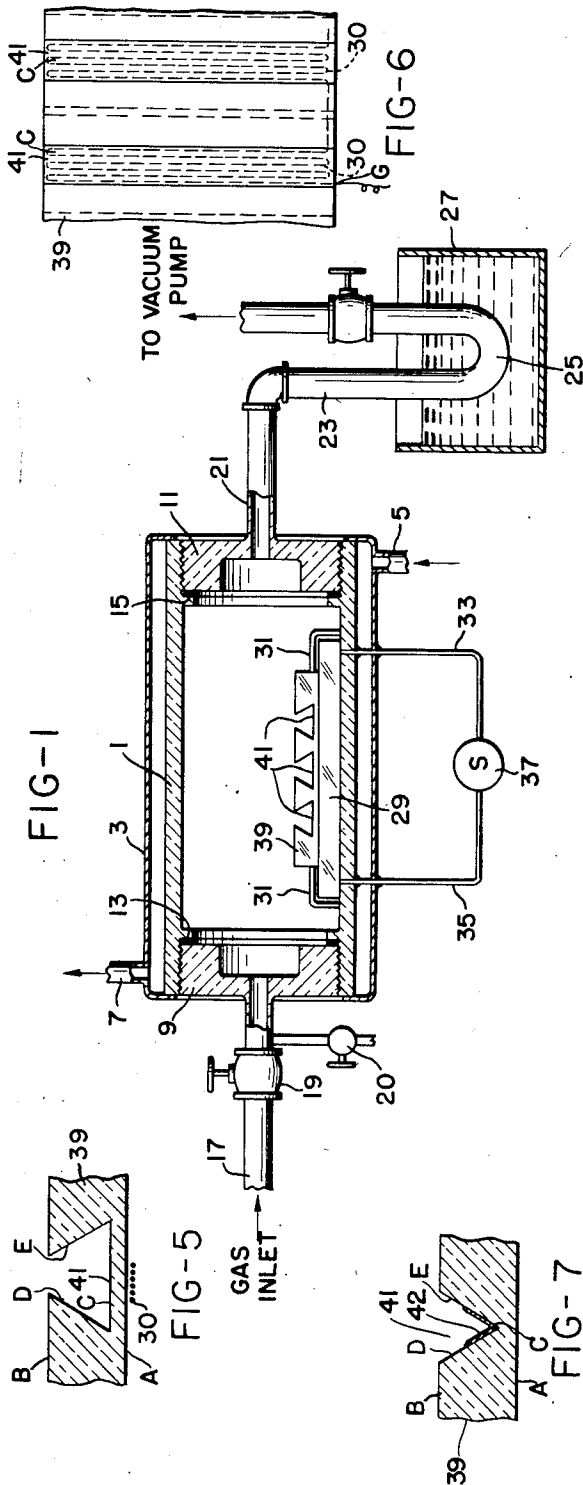


FIG-4

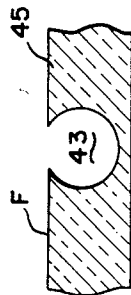


FIG-3

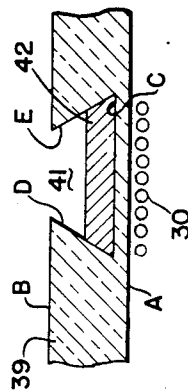


FIG-2

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Application March 18, 1954, Serial No. 417,042

6 Claims. (Cl. 117—212)

This invention relates to the production of electrical resistance heating elements by the thermal decomposition of heat decomposable metal bearing compounds.

This application is related to my copending application Serial No. 417,041, filed March 18, 1954, now Patent No. 2,744,987, issued May 8, 1956, assigned to the same assignee as the present invention.

The invention particularly contemplates the provision of a novel method for the production of metallic films over portions of a body of insulating material. The method involves the deposition of metal from gaseous metal bearing compounds in such manner that only those areas of the body desired to be coated receive metal while those areas immediately adjacent the coated areas are unplated. Such is accomplished in the method of invention without masking of the insulating material.

A further object of the invention is to describe a novel resistance heating element producible with the method of invention.

These and other allied objects of the invention are attained by grooving an electrically non-conductive material to provide a groove base of thin wall section, applying heat to the base locally in the area of the thin wall section in such manner that substantially only the base of the groove is heated to the decomposition temperature of a heat decomposable gaseous metal bearing compound, contacting the material with such a heat decomposable compound to effect deposition in the groove, and then removing the electrically non-conductive material from the plating apparatus with metal deposited substantially only in the groove. The heating of the material is so effected that the material does not attain thermal equilibrium during the plating process; however, a considerable deposition of the metal in the groove is attainable as the deposited metal itself after initial plating thereof on the base portion of the groove conducts heat, thus permitting maintenance of appropriate temperature conditions at the desired area and providing for further metallic deposition.

The invention will be more fully understood by reference to the following detailed description and accompanying drawings wherein:

Figure 1 illustrates an apparatus arrangement useful in the practice of the invention;

Figure 2 illustrates in enlarged fragmentary view one type of electrical resistance element producible with the apparatus of Figure 1;

Figure 3 illustrates in enlarged fragmentary sectional view another and novel resistance heating element;

Figure 4 is a plan view of a product of the invention;

Figure 5 is an enlarged view in vertical section of a portion of the structure of Figure 1;

Figure 6 is a plan view of the structure set out in Figure 5; and

Figure 7 is a view of a further embodiment of an electrical resistor of invention.

Referring to the drawings there is shown in Figure 1 a glass vessel 1 surrounded by a water jacket 3 having

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inlet 5 and outlet 7 and adapted to maintain the interior walls of the vessel cool. Vessel 1 has closing portions 9, 11 tightly and removably sealed thereto with the aid of gaskets 13, 15.

Closing portion 9, has, preferably integral therewith, a gas inlet line 17 having valve 19 which is adapted when opened to permit a flow of plating gas therethrough to the interior of vessel 1.

Closing portion 11 has, also preferably integral therewith, a gas outlet line 21 for the passage of gases outwardly from the vessel 1. This line 21 communicates through a suitable L-shaped coupling with conduit 23 which terminates in a U-shaped portion 25 which extends through a cooling liquid, as Dry Ice and acetone, in trap 27. A vacuum pump (not shown) is adapted to produce a vacuum in the apparatus and to draw exhaust gases to the trap.

Positioned in vessel 1 on the base thereof is an electrical resistance heater 29 partially covered with insulating material 31 and having concentrated heating elements 30 provided with electrical energy through insulated leads 33, 35 (Figure 1) and a suitable source of power 37.

An electrically non-conductive ceramic body 39 is so arranged on heater 29 that the deeply undercut grooves thereon have their bases positioned directly over the concentration of the heating element 30 (Figure 5) the remainder of the ceramic body being substantially unheated for the practice of the invention. In the present illustrative example the ceramic body may be considered to have an overall thickness of 0.5 inch and the thickness of the portion over the element 30 may be about 0.1 inch or about 80 percent of the thickness of the ceramic body has been cut or etched away in the grooving process. The temperature of the heating elements themselves may be maintained at about 450–500° F. and the base of the grooved material will thus be heated very quickly to the temperature of decomposition of, for example, nickel carbonyl, the material which is employed as the plating gas in the present example.

In the practice of the invention with the ceramic piece positioned (Figures 1 and 5) the valve 19 is closed and the apparatus exhausted of all air and gases by operation of the vacuum pump. Heater 29 is at this time operated to assist the volatilization and evacuation of the gases.

After evacuation the valve 19 is opened to admit the nickel carbonyl plating gas. Other carbonyls may be used as the plating gas, such for example as chromium hexacarbonyl, or the gas may be copper acetylacetonate, iron pentacarbonyl, a metal hydride or any metal bearing heat decomposable gaseous compound whose decomposition temperature is sufficiently low to not affect the glass components of the apparatus. If materials having a high decomposition temperature are employed the glass should be replaced by metal and the insulation 31 selected for the temperature conditions.

The entering gas, which for purposes of explanation has been assumed to be nickel carbonyl having a decomposition temperature in the range of about 350–450° F., flows over the ceramic piece 39. This piece has meanwhile been heated by heater 29 the latter being controlled to provide a temperature of 350–450° F. at C (Figure 2) in the bottom of the groove of the ceramic piece. The ceramic piece at the area of the heating element at A (Figure 2) will approach the temperature of the heating element. However due to the relatively poor heat conductivity of the insulating ceramic material and due to the tendency of the surface B to radiate heat into the atmosphere of the vessel 1, the surface B and the upper side wall portions D and E of the groove will not attain the decomposition temperature of the metal bearing carbonyl. The base of the groove will in the case of grooves such as shown in Figures 2 and 3 be heated also due to

radiation from the walls of the groove tending to maintain the base at materially a higher temperature than the surrounding insulating body.

Accordingly surface B which is exposed to the entering gas will be below the decomposition temperature while surface C is above it; no decomposition then occurs at B but considerable deposition takes place at C upon contact of the nickel carbonyl therewith and some slight amount of deposition occurs at the lower portions of wall surfaces D and E.

After deposition is initiated the deposited metal itself of course is highly heat conductive and the upper metal surface temperature will be substantially that of the bottom of the groove, accordingly deposition proceeds readily to the desired extent.

The valve 19 is closed to terminate the plating process when a sufficient deposit of metal 42 has been attained, usually not more than 2 mils in thickness, although the nature of the metal deposited and the characteristic of the service for which a particular resistor is required determine this thickness factor and the invention is not to be considered as limited in this particular respect.

In the passage of the gas then decomposition occurs only in the grooves and as indicated in Figure 4 the resultant product is a grooved body of heat resistant insulating material the plurality of grooves of which are partially filled with metal.

In Figure 3 the groove 43 is shown to have a curvilinear cross-section—preferably formed by reaming out the insulating material 45. The groove is much wider on the interior portion remote from the surface F and is shaped to concentrate the heat within the groove.

The resistor element structure shown in Figure 6 is attainable readily also in the practice of the invention and as illustrated in the figure the deposition of metal may be terminated while the metal itself coats only the side walls, the metal film being in this case in the shape of an open V; such V of course may be closed by continuing metal deposition if desired.

As a further specific example of the practice of the invention chromium carbonyl which decomposes initially at about 375° F. when pure, may be led into vessel 1 with the ceramic piece positioned therein. The temperature of heater 29 may suitably be 475–525° F. and the ceramic piece would then have a temperature in the base of the groove of between about 375–425° F., while the remainder of the body is substantially cool. The interior groove surfaces of a piece such as that shown in Figure 2 or Figure 3 would then be in the decomposition range and deposition would occur readily while the upper surface is free of metal.

The operation of the process is predicated then upon the development in the groove of insulating material of a temperature sufficient to effect decomposition, while maintaining the remainder of the piece below the decomposition point; this eliminates the necessity for removing the material after plating or for masking the material prior to plating, which is sometimes particularly difficult with elements of complicated shape.

It is to be noted that the characteristics of the material plated are thus utilized to inhibit the attainment over portions of the material plated of a temperature sufficient to occasion decomposition; the nature of the material itself, the particular temperature range employed for plating and the required characteristics of the product vary over wide ranges, and there follows a chart illustrating the preferred conditions of use of material generally suitably employable in the production of the product of this invention. It will, in general, be found that where the plating material is ceramic or glass and has an overall thickness of $\frac{3}{8}$ of an inch or more and a groove depth of at least 80 percent of the thickness of the object, it is most suitable for plating under the conditions set out. It is understood that this chart is not limited but merely exemplary of the use and conditions:

Plating Material	System Pressure, mm. of Hg	Temperature Range of Base Material in Degrees Fahrenheit
Ni(CO) ₄	0.5-1.0	350-450
Cr(CO) ₃	0.5-1.8	375-450
Mo(CO) ₆	0.5-1.8	750-850
W(CO) ₆	0.5-1.8	625-775
Cu(C ₂ H ₄ O) ₂	0.5-1.8	400-750

The product of the invention will not only be plated in the groove but the edge, such as at G in Figure 4, between the groove and the lowermost surface of the insulating body may be desirably plated by exposing the same to the metal bearing gases; such may then be utilized as terminal points of the electrical resistor if desired. Since each groove will have a plated edge associated therewith a considerable range of resistance values may be attained with one resistor depending upon the number of grooves and the point of connection of the leads to the resistor.

The product is particularly advantageous since the electrically conductive material in film form, which is highly resistant to the passage of current, will be protected from mechanical damage due to its recessed position.

It will be understood that this invention is susceptible to modification in order to adopt it to different usages and conditions and accordingly, it is desired to comprehend such modifications within this invention as may fall within the scope of the appended claims.

I claim:

1. In the process of producing an electrical resistor the steps of selectively heating a localized base portion of an electrically non-conductive and poorly heat conductive body adjacent a groove of the body to raise the temperature of the groove base to that of the decomposition temperature of a gaseous heat decomposable compound, and contacting the body with a gaseous metal bearing compound decomposable at the temperature of the groove base but not at the temperature of the surrounding body to effect deposition of metal on the groove base and in the groove, said heating being restricted to only the bottom of said groove while the remainder of the body is substantially cool.

2. In the process of producing an electrical resistor the steps of selectively heating a localized base groove portion of an electrically non-conductive and poorly heat conductive body to a temperature at which a heat decomposable gaseous metal compound decomposes the heating taking place at a rate such that substantially only the localized portion is heated to the decomposition point, and contacting the body with a gaseous metal compound decomposable at the temperature of the base groove portion to effect metallic deposition therein, said heating being restricted to only the bottom of said groove while the remainder of the body is substantially cool.

3. The method of producing electrical resistors which comprises the steps of grooving a thickness of an electrically non-conductive material to an extent such that the heat transfer through the material remaining ungrooved and defining the base of the groove is substantially greater than that of the heat transfer through the material in its ungrooved thickness, heating the material only at the area adjacent the base of the groove to a temperature sufficient to decompose a heat-decomposable metal bearing gaseous compound, and then contacting the electrically non-conductive material with said heat decomposable metal bearing gaseous compound while substantially only the base of the material is at a temperature as high as the decomposition temperature of the heat decomposable metal bearing gas.

4. In a gas plating process for the production of electrical resistors, the steps of locally heating a thin groove base section of an electrically non-conductive body of material

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to an extent such that the portion locally heated is raised to at least the thermal decomposition point of a heat decomposable metal bearing compound while other thicker portions of the body around the groove are lower in temperature than the said decomposition temperature, contacting the body with a gaseous metal bearing compound which is decomposable at the temperature of the locally heated portion of the body to deposit metal on the body, and then removing the body from the heat and the gaseous contact, with the metal deposited on the body only in the locally heated portion, said heating being restricted to only the bottom of said groove while the remainder of the body is substantially cool.

5. The method of producing electrical resistors which comprises the steps of grooving a thickness of an electrically non-conductive and poorly heat conductive material to the extent of at least 80 percent of the thickness of the material and such that the heat transfer characteristic of the material remaining ungrooved and defining the base of the groove is substantially greater than the heat transfer through the material in its ungrooved thickness, confining the heating of the material only at the bottom of the groove to a temperature in the range of about 200°-1000° F., and then contacting the electrically non-conductive, poorly heat conductive material with a metal bearing gaseous compound which is decomposable thermally in the range of 200°-1000° F. and while substantially only the material in the bottom of said groove is at a temperature as high as the decomposition temperature of the heat decomposable metal bearing gaseous compound.

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6. A process of producing an electrical resistor which comprises providing a grooved body of an electrically non-conducting ceramic material of such heat transmission characteristics that a thin section thereof may be quickly heated to a temperature sufficient to effect thermal decomposition of a heat decomposable compound without material transmission of heat through the body from the thin section, heating a thin section bounding a groove of the body to the thermal decomposition temperature of a heat decomposable compound without substantially heating the remaining portion of the body, while the thin section only is at the decomposition temperature contacting the body with a gaseous metal bearing compound decomposable at that temperature to deposit metal on the section, and removing the body from the heat and gas before the remaining portion of the body has attained the decomposition temperature of the heat decomposable compound, said heating being restricted to only the bottom of said groove while the remainder of the body is substantially cool.

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