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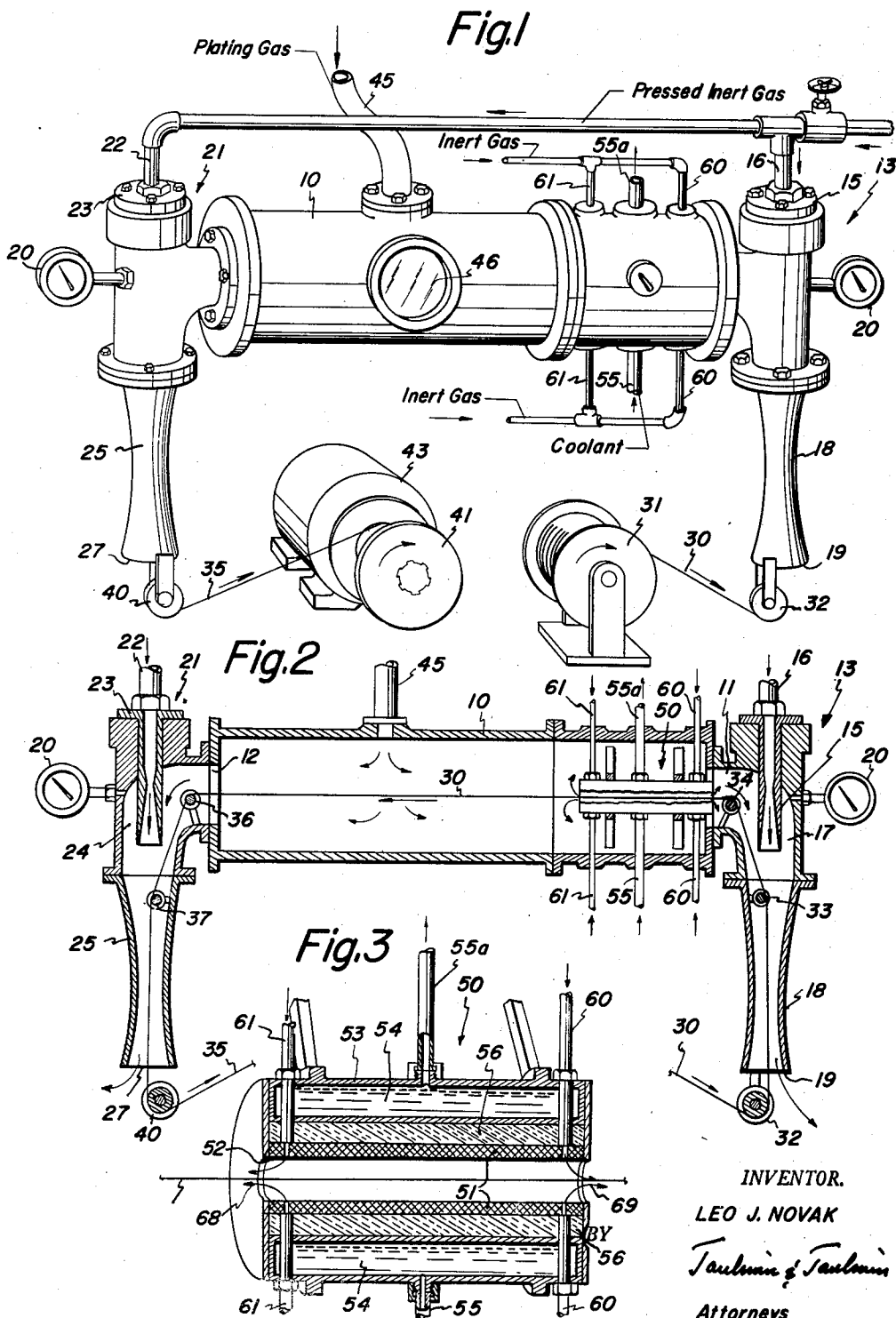
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CONTINUOUS GAS PLATING APPARATUS UNDER VACUUM SEAL

Filed March 9, 1956

2 Sheets-Sheet 1



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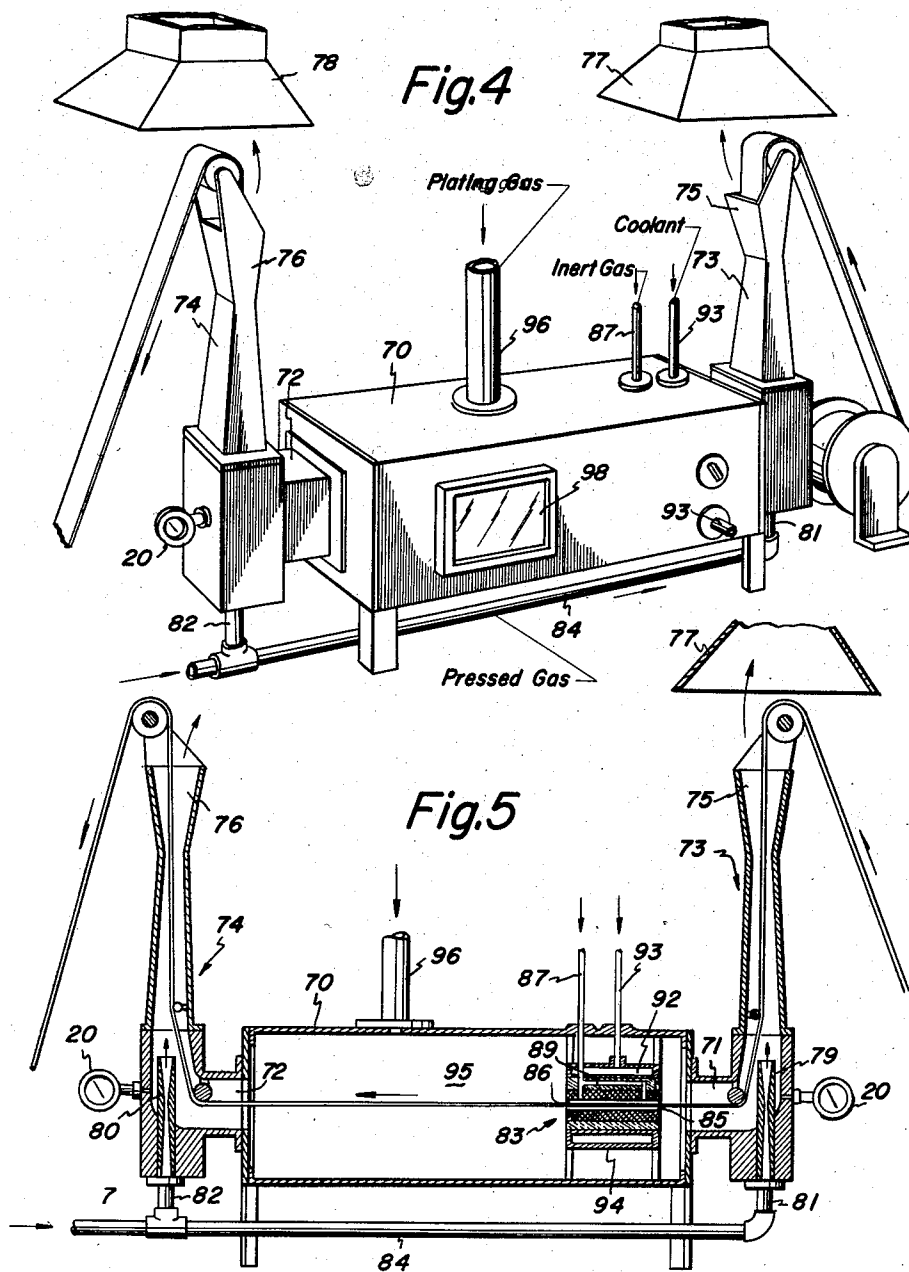
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CONTINUOUS GAS PLATING APPARATUS UNDER VACUUM SEAL

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CONTINUOUS GAS PLATING APPARATUS
UNDER VACUUM SEAL

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1 Claim. (Cl. 118—49)

This invention relates to the art of deposition of metals by gas plating. More particularly, the invention appertains to an apparatus and method for gas plating long continuous length strips, fibers or filaments of organic, inorganic, metallic, or non-metallic material.

The invention provides an apparatus which permits gas plating of continuous length material in plating chamber maintained at a pressure below atmospheric, and wherein both the inlet and outlet of the plating chamber is sealed by an ejector type pump or aspirator.

An object of this invention is to provide an apparatus and method for continuously gas plating materials moved through a plating chamber in which the pressure is maintained below atmospheric by the use of suction pumps connected to the inlet and outlet openings of the plating chamber, and without hindering the movement of the material to be plated through the plating chamber.

Another object of the invention is to provide an improved apparatus and method for carrying out gas plating which includes an elongated plating chamber open at both ends for the passage of material into and out of the plating chamber while creating suction at the openings to the chamber.

Another object of the invention is to provide an apparatus and method for gas plating articles which are disposed in a plating chamber or enclosure having inlet and outlet openings through which the article to be plated is moved while said openings are fluid sealed against the entrance of air or surrounding atmosphere by means of ejector pumps installed at said openings.

Still another object of this invention is to provide a vacuum gas plating system whereby one can use organo-metallic materials which are otherwise relatively non-volatilizable at ordinary atmospheric pressures, as for example molybdenum carbonyl, and wherein the gas plating is carried out as a continuous process.

These and other objects and advantages of the invention will be apparent to those skilled in the art from the following description taken in conjunction with the accompanying drawings wherein two apparatus embodiments are illustrated which are suitable for practicing the invention.

In the drawings:

Figure 1 depicts pictorially a plating apparatus for carrying out the gas plating of filaments or long continuous length strands of material in accordance with this invention;

Figure 2 is a vertical section taken of the gas plating apparatus shown in Figure 1;

Figure 3 is a perspective view and illustrating in longitudinal section and on an enlarged scale the construction of the heating element arranged in the plating chamber;

Figure 4 illustrates in perspective a modification of the embodiment shown in Figures 1 and 2, and wherein the inlet and outlet openings to the plating chamber open upwardly and beneath a hood, a continuous length band or strip being illustrated as the material being gas plated; and

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Figure 5 is a vertical section taken through the apparatus illustrated in Figure 4.

The gas plating apparatus of the invention embodies the principles of aspiration and provides a plating chamber having its inlet and outlet openings through which material to be gas plated enters and exits, fluid sealed by inert gas ejected at high velocity across the inlet and outlet to the chamber while the material being gas plated is heated and moved through the plating chamber. Heating of the material to be plated takes place inside the plating chamber, the material being quickly heated to a temperature high enough to bring about decomposition of the gaseous metal plating compound introduced into the plating chamber and circulated into contact with the material to be gas plated and which is flash heated upon entering the plating chamber.

In the apparatus illustrated in Figures 1-3, a plating chamber is illustrated at 10, which is cylindrical in cross section and provided with an inlet 11 and an outlet 12 at its opposite ends as best illustrated in Figure 2. Positioned at the inlet opening 11 and communicating therewith is an ejector type pump or fluid aspirator, generally designated 13. This ejector pump is of the usual construction having a nozzle jet 15 through which fluid such as air, carbon dioxide, helium, argon or the like is pumped or flows at high velocity, being introduced through pipe 16. The fluid flows, as illustrated by the arrows in Figure 2, from the nozzle 15 extending into the inlet chamber portion 17 which communicates with the inlet 11, and outward through the venturi-shaped nozzle 18 and is discharged through the opening 19.

As the fluid flows at high velocity through the venturi nozzle 18, it develops a suction which causes some of the gas in the plating chamber 10 to be drawn into the venturi nozzle and thus create a partial vacuum or sub-atmosphere pressure conditions in the plating chamber. To indicate the pressure in the inlet chamber portion 17, a gauge 20 is provided, as illustrated in Figure 2.

At the opposite end of the gas plating chamber 10 there is provided a second ejector type jet pump, generally designated 21. This pump is of similar construction and operation as the ejector 13. Fluid under pressure is admitted through the conduit 22 to the nozzle jet 23 which extends into the chamber portion 24 which communicates with outlet 12 of the plating chamber. Fluid is discharged from nozzle 23 and passes through the venturi-shaped nozzle 25 and exits as at 27. Operation of the ejector pumps is carried out simultaneously while the material, generally indicated at 30, is moved through the plating chamber so that sub-atmospheric pressure conditions are maintained in the plating chamber. The inlet and outlet openings 11 and 12 are effectively sealed during operation of the ejectors 13 and 21 by reason of the pressure set up by high velocity gas flowing through the venturi nozzles 18 and 25.

When gas plating material, as by the use of a metal carbonyl, the filament or long continuous length material, such as illustrated at 30 in Figure 1, is drawn from a storage roll 31 and passed around a guide roll 32 arranged at the discharge opening 19 of nozzle 18. From guide roll 32 the filament 30 is carried upwardly through the nozzle 18, and over the rollers 33 and 34, and therealong through the plating chamber 10 and out through the outlet opening 12. The plated filament 35 then passes over guide rolls 36 and 37 and outwardly through the venturi nozzle 25 and opening 27. The gas plated material 35 is guided around roll 40 and is finally wound up on roll 41, or if desired, the plated material may be passed directly to a fabricating machine such as a felting, weaving apparatus.

The filament 30, as illustrated in Figure 1, is suitably drawn from the storage roll 31 and passed through the

plating chamber 10 by a motor 43 which is connected to drive the roll 41. Motor 43 is driven at a controlled speed so that the filament is drawn from the storage roll 31 and moved through the plating chamber at a uniform speed which may be varied using suitable speed control means, not shown, whereby gas plating different kinds of material may be accomplished.

Operation of the ejectors 13 and 21 functions to set up a vacuum or sub-atmospheric pressure conditions in the interior of the plating chamber and concurrently fluid seal the inlet and outlet openings against in-flow of gases from the surrounding atmosphere to the plating chamber. A heat-decomposable gaseous metal compound, and such as commonly used for gas plating, is introduced into the plating chamber by conduit 45 the latter being preferably connected centrally with the plating chamber, as illustrated in Figures 1 and 2. In this manner the plating gas is evenly distributed throughout the plating chamber.

Metal plating gas flows uniformly towards the inlet and outlet openings of the plating chamber, as illustrated by the arrows in Figure 2 upon actuation of the jet pumps to set up substantially equal suction pressures, as indicated by gauges 20 located at the inlet and outlet openings. A window 46 in the wall of the plating chamber is provided for viewing the plating operation.

For heating the filament or material being gas plated, there is provided a heater generally designated 50, the construction of which is illustrated in Figure 3. The heater comprises an elongated open coil member 51 having a central opening 52 extending therethrough. The material to be plated is adapted to be passed centrally through the opening 52, the heater being suitably positioned adjacent the inlet opening 11 so that the filament or material to be gas plated is heated immediately upon entering the plating chamber and before it is brought into contact with the plating gas.

The heater 50, as illustrated in Figure 3, comprises an outer casing 53 which is provided with a jacket or shell portion 54 through which cooling fluid or liquid, such as water, is circulated to maintain the outer casing wall below the temperature at which the metal plating gas will heat decompose. Cooling fluid is suitably introduced to the jacket by means of inlet and outlet conduits 55 and 55a respectively. Arranged around and adjacent the cooling jacket 54 is an insulating wall 56 which is preferably made of glass or mineral wool. A heating element or open coil 51 comprises a concentrically disposed asbestos resistance wire or ribbon suitably wound therearound and connected to a source of electric current.

To prevent passage of the heat-decomposable gaseous metal plating compound into the interior of the heating coil or chamber 51, inert gas is forced into the inlet and outlet openings to the chamber through suitable conduits 60 and 61 disposed at the inlet and outlet openings of the heater coil. In this way inert gas is caused to flow into the inlet and outlet openings of the heater, as illustrated by the arrows in Figure 3, and to create a pressure slightly above that of the interior of the plating chamber 10 so that the plating gas is prevented from entering the heater 50.

In the use of the apparatus, as illustrated in Figures 1-3, gas such as air, steam, nitrogen, argon or helium, is connected for operating the jet pumps arranged at opposite ends of the plating chamber. This gas or fluid for operating the ejectors is introduced under a pressure of two to three hundred pounds per square inch, being controlled by suitable valves, not shown, to provide the desired suction pressure. Filament material which is to be plated is preferably drawn into the plating chamber in opposite direction to that of the gas as the filament is moved through the inlet and into the plating chamber. The discharge nozzle of the ejector pump is suitably arranged so that the filament enters against the outward flow of ejector gas from inlet nozzle 18 while in the same direction of gas flowing from the exit nozzle

25. The material thus to be plated is passed through the first ejector 13 then through the heating element 50 and through the plating chamber 10, and finally exits from the plating chamber through the ejector pump 21, the plated material being drawn out through the ejector pump in the same direction as the flowing ejector gases and wound up on a storage roll.

As illustrated in Figure 3, inert gas flowing through the conduits 60 and 61 is discharged adjacent the inlet and outlet opening and flows outwardly, as illustrated by the arrows 63 and 69 in Figure 3. In this manner, an effective gas seal is provided at the inlet and outlet openings to the heater and thus prevents the heat-decomposable gaseous metal compound used for plating from entering the heating coil and thus prevents decomposition of the thermally labile metal containing gases in the plating chamber should they come into contact with the heater coils. A chemically inert gas suitable for this purpose is helium, argon, carbon monoxide or dioxide or nitrogen. The inert sealing gas preferably is preheated to a temperature approximating that at which the gaseous metal compound will decompose. This inert gas is introduced under sufficient pressure so that it flows outwardly at both the inlet and outlet ends of the heater, as described, to thus maintain the heater free of gas plating compound.

Gas plating may be carried out simultaneously on one or more fibers, as for example glass fibers, filaments or threads, as well as other materials such as nylon or metal wire, by moving the material through the ejectors and plating chamber as illustrated in the drawings, the ejectors being operated along with heater sealing gas seals so that a continuous seal is provided in the plating and heater chambers during gas plating.

An important feature of the invention consists in controlling the rate of gas issuing from each of the heater orifices as shown in Figure 3, whereby the entrance of gaseous metal plating gas into the heater is prevented. Another important feature resides in maintaining the outer walls of the heater which come in contact with metal plating gas a few degrees (5-10° F.) cooler than the temperature at which the gaseous plating compound decomposes in the plating chamber. This is readily accomplished by circulating cold water or cooling fluid through the outer jacket 54 of the heater 50, as illustrated in Figure 3. In this way the outer walls of the casing 53 are maintained at a temperature such that gas plating of the heater casing does not take place. On the other hand, the inner opening or core of the heater and through which the filament passes to heat the same is maintained at a relatively high temperature and such as will quickly heat the material to be plated. A flash heating of the filament or fiber is brought about as it is moved along continuously through the heater and thence through the gas plating chamber proper and in which the resultant heated material is brought into contact with the heat-decomposable gaseous metal compound while heated to a temperature which brings about decomposition of the gaseous plating compound and deposition of the metal constituent onto the filament or fiber material.

Referring to Figures 4 and 5, wherein a modification of the apparatus shown in Figures 1-3 is illustrated, a rectangular plating chamber 70 is shown having inlet and outlet openings 71 and 72 respectively, for passage of material to be gas plated.

In the apparatus illustrated, ejector fluid pumps 73 and 74 are suitably mounted at the inlet and outlet openings respectively of the plating chamber 70, the same being of similar construction and operation as ejector pumps 13 and 21 of the apparatus illustrated in Figures 1 and 2. In the gas plating structure illustrated in Figures 4 and 5, the venturi discharge nozzles 75 and 76 of the ejectors are disposed upwardly and arranged to discharge waste gas into a hood or the like as indicated at 77 and 78 in Figure 4. Fluid or gas for operating the ejector pumps 73 and 74 is admitted to the jets 79 and 80

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through pipe sections 81 and 82 respectively, which in turn, are connected to a common supply conduit 84 as illustrated in Figure 5. Gas for operating the ejector pumps flows through the jets as illustrated by the arrows in Figures 4 and 5.

A heater 83 is provided adjacent to and communicating with opening 71 of the plating chamber 70. Inert gas is introduced into the heater and adjacent to the inlet opening 85 and outlet opening 86 to fluid seal these openings against ingress of plating gas into the heater. Conduit 87 is provided for passing gas to the common pipe 89 whereby inert gas is discharged in the heater and adjacent the inlet and outlet thereto.

Cooling fluid is admitted to the outer jacket 92 through suitable conduits 93 to maintain the outer walls or casing 94 cool. Gas plating gas is suitably admitted to the interior of the plating chamber as at 95, by means of a conduit 96.

The construction and operation of the heater 83 is the same as heater 50 which has been described.

An observation window 98 is provided for viewing the interior of the plating chamber when desired. The operation of the modified gas plating apparatus shown in Figures 4 and 5 is similar to that illustrated and described in Figures 1-3 and will be readily understood without further description.

An example of the gas plating conditions employed in gas plating glass filament as drawn from a molten mass of glass with nickel metal and employing nickel carbonyl is illustrated by the following—

Pressure in gas plating chamber..... 10 mm. Hg.
 Temperature in heater chamber..... 500° F.
 Rate of flow of gaseous $\text{Ni}(\text{CO})_4$ 0.25 cu. ft./min.
 Ejector gas line pressures (inlet and outlet)..... 100 lbs./sq. in.
 Flow of inert gas through heater..... 0.1 cu. ft.
 Cold water inlet temperature to heater..... 85° F. maximum.
 Rate of glass filament feed..... 125 ft./min.

Different metals may be gas plated employing the apparatus and method described. Metals other than nickel as mentioned may be gas plated on any material, as desired, for example carbonyls of chromium, iron, tungsten, molybdenum may be used as the heat-decomposable compound to deposit the desired metal.

Metals to be deposited may be introduced as gaseous metal carbonyls or vaporized solutions of certain of the metal carbonyls in readily vaporizable solvents (for example, petroleum ether), also nitroxyl compounds, nitrosyl carbonyls, metal hydrides, metal alkyls, metal halides, and the like.

Illustrative compounds of the carbonyl type are nickel, iron, chromium, molybdenum, cobalt, and mixed carbonyls. Other compounds which may be used are the nitroxyls, such as nickel and copper nitroxyls; nitrosyl carbonyls, cobalt nitrosyl carbonyl; and hydrides, such as antimony hydride, tin hydride; and metal alkyls, such

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as aluminum and magnesium alkyls, and halogens, for example, chromyl chloride, osmium carbonyl bromide, ruthenium carbonyl chloride, as well as acetates, e. g., copper acetylacetonate.

Each material from which a metal may be plated has a temperature at which decomposition is complete. However, decomposition may take place slowly at a lower temperature or while the vapors are being raised in temperature through some particular range. For example, nickel carbonyl completely decomposes at a temperature in the range of 375° F. to 400° F. However, nickel carbonyl starts to decompose slowly at about 175° F. and therefore decomposition continues during the time of heating from 200° F. to 380° F.

A large number of the metal carbonyls and hydrides may be effectively and efficiently decomposed at a temperature in the range of 350° F. to 450° F. When working with most metal carbonyls we prefer to operate in a temperature range of 375° F. to 425° F.

It will be understood that while the method and apparatus disclosed and described herein illustrate a preferred form of the invention, modification can be made without departing from the spirit of the invention, and that all modifications that fall within the scope of the appended claim are intended to be included herein.

What is claimed is:

A gas plating apparatus for gas plating metal onto the surface of long continuous length material by thermal decomposition of a gaseous metal bearing compound brought in contact therewith, said apparatus comprising an elongated plating chamber having an inlet at one end and an outlet at the other end through which the material to be gas plated is moved therealong through said plating chamber, means comprising a heating element arranged in said plating chamber and adjacent the inlet thereof, means for introducing a thermally decomposable gaseous metal bearing compound into the plating chamber, an ejector fluid actuated pump disposed at said inlet, an ejector fluid actuated pump disposed at said outlet, venturi-shaped nozzle means arranged about both the inlet and the outlet of the plating chamber and coacting with the ejector pump to receive and discharge fluid from said pump at high velocity at said inlet and outlet for fluid sealing the same against ingress of gases from the surrounding atmosphere, and spaced guide roll means arranged in said inlet and outlet at opposite ends of said plating chamber to guide the continuous length material through the venturi-shaped nozzles and centrally through the heating element and said elongated plating chamber.

References Cited in the file of this patent

UNITED STATES PATENTS

1,710,747	Smith	Apr. 30, 1929
2,332,309	Drummond	Oct. 19, 1943
2,656,284	Toulmin	Oct. 20, 1953
2,657,457	Toulmin	Nov. 3, 1953
2,749,255	Nack et al.	June 5, 1956