

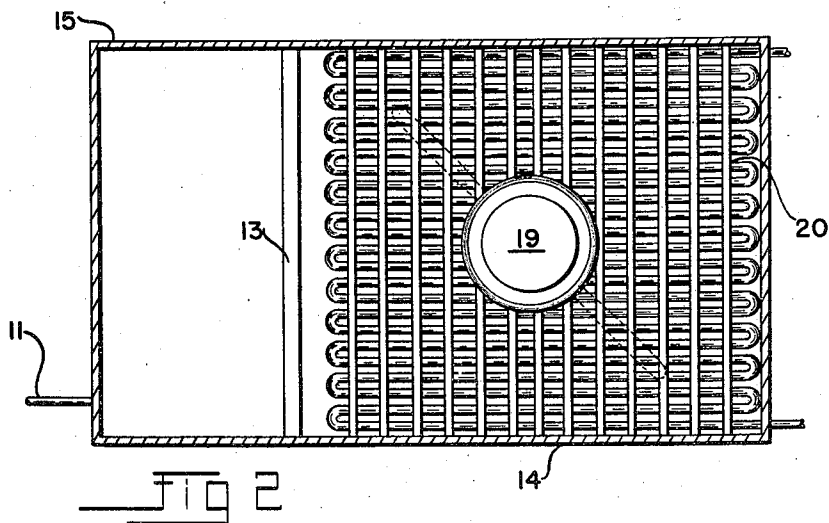
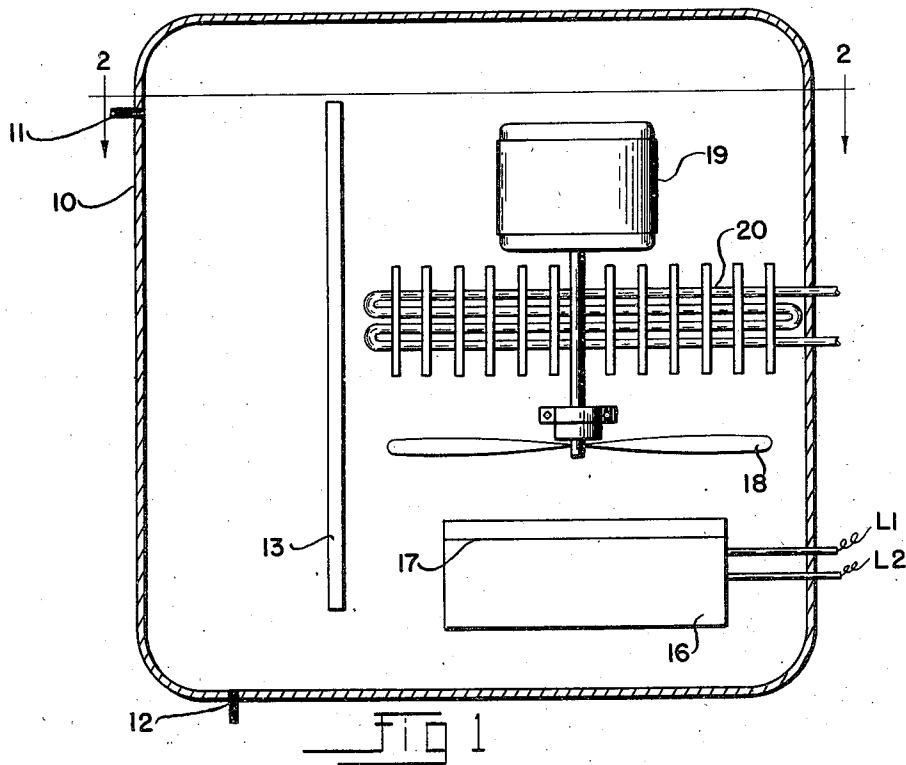
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DYNAMIC PYROLYTIC PLATING PROCESS

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DYNAMIC PYROLYTIC PLATING PROCESS

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This invention relates to the art of deposition of metals. More particularly it relates to the plating of metals from the gaseous state and the apparatus for carrying out the process.

Deposition of thin films of protective metal such as nickel, cobalt, tungsten, molybdenum, their metal alloys, and the like, has been accomplished in the past by enclosing an object to be plated in a chamber, charging the chamber with decomposable metal-bearing gas, and heating the object to the decomposition temperature for said gas.

In ordinary pyrolytic plating, the production of dense metal deposits is made difficult by two phenomena, namely, intergaseous decomposition and gas film resistance to true surface contact.

Intergaseous decomposition is caused by the plating gas becoming heated to an undesirably high temperature while not in contact with the object to be plated. This phenomenon causes particles of the metal to be formed and which fall on the plating surface, producing extreme roughness. Obviously, for sound plating this phenomenon must not be permitted to occur.

The second phenomenon, gas film resistance, is always present in gas to heated-solids systems and serves as a boundary between the solid and the gas. It prevents the gaseous metal compound from decomposing directly on the free surface of the metal.

The results of such phenomena show themselves in the form of rough irregular plates, and thin porous coating.

It is an object of the present invention to overcome the disadvantages and limitations of the above described methods.

It is another object of the present invention to provide a method which increases the density of the coating.

It is a further object of the present invention to provide a method which gives metal coatings in decreased porosity.

It is a still further object of the present invention to provide a method which produces increased smoothness and brightness of deposit.

It is still another object of this invention to provide a method having increased efficiency due to elimination of intergaseous decompositions and the deposit of loose metal powder in the bottom of the plating chamber.

It is also an object of the present invention to provide a method for producing increased density deposits adaptable to discontinuous or continuous plating.

It is also an object of the present invention

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to provide apparatus for carrying out the above processes.

These and other more specific objects will become apparent from the following description:

The plating as carried out in accordance with this invention is keeping the gas temperature below the intergaseous decomposition point and directing the metal-bearing gases in agitated, turbulent flow against the surface to be plated.

In this process, decomposable metal-bearing gases are heated to the decomposition temperature by heat radiating from the object being plated. The hot but undecomposed gases are recirculated through the system. Gas velocities and volumes maintained within the plating chamber quickly bring the hot gases into contact with a cooler, whereby a gas temperature in the range of 110° F. to 175° F., and preferably about 125° F., is continuously maintained.

To maintain the concentration of metal-bearing gases, fresh gases at a temperature of about 100 F. are continuously or intermittently bled into the gases being recirculated in the chamber.

The gaseous atmosphere may be formed by mixing an inert gas with the vapors of a volatile metal compound, or by atomizing a liquid metal compound into a blast of warm inert gas or other equivalent method.

Carbon dioxide, helium, nitrogen, hydrogen, the gaseous product of controlled burning of hydrocarbon gases free of oxygen, and the like, have been utilized as a carrier medium or inert gas medium.

In some instances the use of hydrogen is preferred, as for example, in a cleaning anneal chamber where its ability to act as a reducing agent may be put to advantage to remove the oxide film or rust from iron.

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.

Illustrative compounds of other groups are the nitroxyls, such as copper nitroxyl; nitrosyl carbonyls, for example, cobalt nitrosyl carbonyl; hydrides, such as antimony hydride, tin hydride; metal alkyls, such as chromyl chloride; and carbonyls-halogens; for example, osmium carbonyl

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bromide, ruthenium carbonyl chloride, and the like.

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 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 I prefer to operate in a temperature range of from 375° F. to 425° F.

Heating of objects to temperatures in the above range may be accomplished in many ways depending upon the type of object being plated. If the object is stationary, the object may be set on a resistance heater or so-called hot plate. If the object is moving, it may be heated by passing over resistance heaters, or by infra red light, or by passage of electricity therethrough either of standard or high frequency or like means.

Preparatory to coating the material may be cleaned by employing conventional methods used in the art, comprising electro-chemically cleaning by moving the same through a bath of alkali or acid electrolyte in which the object is made the cathode or anode.

Pickling with hydrochloric, sulfuric or nitric acid, or a combination of acids, may also be made a part of the cleaning process and the object thoroughly rinsed or washed prior to introduction into the plating apparatus.

The invention will be more clearly understood from the following description of one embodiment of the apparatus and its mode of operation, taken in connection with the drawing wherein:

Figure 1 is a diagrammatic illustration of a complete plating unit; and

Figure 2 is a top view along the line 2—2 of Figure 1.

Referring to the drawing, there is shown a housing 10, which is provided with gas inlet and outlet 11 and 12, respectively. The internal arrangement is illustrated with respect to vertical gas flow within a single chamber, but other arrangements are equally feasible.

Within housing 10 is a vertical panel 13 extending from the front to the back walls 14 and 15. Positioned adjacent the bottom of housing 10 is a heater 16 having a top plate 17 capable of heating metal objects deposited thereon to temperatures in the range of 350° F. to 500° F., preferably 350 to 450° F. Heater 16 receives electric current through the lines L1 and L2.

Above the hot plate 17 is suspended a fan 18 driven by an enclosed motor 19. This fan is adapted to maintain gas velocities of the order of 40 to 90 feet per second.

In the gas recirculation system, is a gas cooler 20 receiving coolant from an external system (not shown).

While this apparatus is shown as contained within a single housing, it will at once be apparent that the cooling of gas may be accomplished outside the plating zone and the gas recirculated by a fan taking suction on the plating

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chamber and forcing the gas back to the chamber through an outside duct.

The operation of this apparatus is illustrated by the following examples:

Example I

Copper discs were wire brushed to clean the surface. The discs were positioned on the hot plate and heated to a temperature of approximately 400° F. Into the housing was fed a mixture of vaporous nickel carbonyl and carbon dioxide, the carbonyl being present in the proportions of approximately 5 ozs. of carbonyl per cubic foot of carbon dioxide.

The copper discs were plated in accordance with standard operating procedure of passing approximately 3 cubic feet of gas mixture per hour through the housing 10, for a period of 2 minutes, at the end of which time a plating of .0012 inch of nickel was obtained.

Example II

New copper discs treated as in the previous operation were positioned on the hot plate and an identical gas mixture fed to the chamber. Inside the chamber the gas was maintained at a temperature of approximately 125° F. by the coolant. However, the gas circulated in the housing 10 at a rate of about 60 feet per second due to the operation of fan 18. The plating time was 2 minutes, as in Example I, and the depth of deposit .0033 inch of nickel.

Upon comparison of the discs, it was found that the deposits obtained by the high gas velocity procedure were smoother. In addition, on the basis of the weight of metal deposited, the density of deposit on samples plated without mechanical agitation was 47 grams per cubic inch, whereas the density of the samples plated employing the fan to cause turbulent gas flow was 128 grams per cubic inch.

In salt tests the discs of Example II did not show signs of corrosion after 2 days, whereas the discs of Example I showed signs of corrosion after 4 hours.

It will be understood that while there have been given herein certain specific examples of the practice of this invention, it is not intended thereby to have this invention limited to or circumscribed by the specific details of materials, proportions, or conditions herein specified, in view of the fact that this invention may be modified according to individual preference or conditions without necessarily departing from the spirit of the disclosure and the scope of the appended claims.

I claim:

1. The method of gas plating dense coatings which comprises: heating an object to a decomposition temperature for metal-bearing vapors in a plating zone; moving gaseous material at least a portion of which decomposes depositing metal into the said plating zone; and moving the gas at a velocity of between about 40 and 90 feet per second in a turbulent fast moving stream directed against the surface of the object to be plated.

2. The method of gas plating dense coatings which comprises: heating an object to a decomposition temperature for metal-bearing vapors in a closed plating zone to a temperature in the range of 350° F. to 450° F.; moving gaseous material at least a portion of which decomposes depositing metal into the said plating zone; and moving the gas at a velocity of between about

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40 and 90 feet per second in a turbulent fast moving stream directed against the surface of the object to be plated.

3. The method of gas plating dense coatings which comprises: heating an object to a decomposition temperature for metal-bearing vapors in a plating zone; feeding a gaseous mixture of metal carbonyl and inert gas into the said plating zone; and moving the gas at a velocity of between 40 and 90 feet per second in a turbulent fast moving stream directed against the surface of the object to be plated.

4. The method of gas plating dense coatings which comprises: heating an object to a decomposition temperature for metal-bearing vapors in a plating zone; feeding a gaseous mixture of nickel carbonyl and carbon dioxide into the said plating zone; and moving the gas at a velocity of between about 40 and 90 feet per second in a turbulent fast moving stream directed against the surface of the object to be plated.

5. The method of gas plating dense coatings which comprises: heating an object to a decomposition temperature in a plating zone; feeding gaseous material at least a portion of which decomposes depositing metal into the said plating zone; recycling the gas passing the heated object and causing it to strike repeatedly under high velocity turbulent flow conditions and moving at a velocity between about 40 and 90 feet per second against the hot surface to be plated; and cooling the recycled stream to temperatures below the intergaseous decomposition range.

6. The method of gas plating dense coatings which comprises: heating an object to a decomposition temperature in a plating zone; feeding gaseous material at least a portion of which decomposes depositing metal into the said plating zone; recycling the gas passing the heated object and causing it to strike repeatedly under high velocity turbulent flow conditions and moving at a velocity between about 40 and 90 feet per second against the hot surface to be plated; and cooling the recycling stream to temperatures in the range of approximately 110° F. to 175° F.

7. The method of gas plating dense coatings which comprises: heating an object to a decomposition temperature in a closed plating zone; feeding gaseous material at least a portion of which decomposes depositing metal into the said plating zone under turbulent flow conditions; recycling the gas within the said plating zone; intermittently enriching the recycled stream with metal bearing vapors; cooling the recycling

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stream to temperatures below the intergaseous decomposition range; and directing the turbulent flow of the mixed cooled stream at a velocity of between about 40 and 90 feet per second against the hot surface to be plated.

8. The method of gas plating dense coatings which comprises: heating copper discs to a temperature of approximately 400° F. in a closed plating zone; feeding a gaseous mixture of nickel carbonyl and carbon dioxide into the plating zone at the rate of 3 cubic feet per hour, said carbonyl ratio being approximately 5 ozs. of carbonyl per cubic foot of carbon dioxide; recycling the gaseous mixture within the plating zone at a rate of between 50 to 500 cubic feet per minute; and cooling the recycling stream to a temperature of approximately 125° F.

9. The method of gas plating dense coatings which comprises: heating steel plates to a temperature of approximately 400° F. in a closed plating zone; feeding a gaseous mixture of molybdenum carbonyl and carbon dioxide into the said plating zone at the rate of 3 cubic feet per hour, said carbonyl ratio being approximately 5 ozs. of carbonyl per cubic foot of carbon dioxide; recycling the gaseous mixture within the plating zone at the rate of between 50 to 500 cubic feet per minute; and cooling the recycling stream to a temperature of approximately 125° F.

10. In the process of gas plating dense coatings the steps of imparting high velocity turbulent flow to a gas stream and directed against a heated object with said gas stream while the gas is moving at a velocity between about 40 and 90 feet per second, and maintaining the ambient gas temperature below the intergaseous decomposition temperature.

11. In the process of gas plating dense coatings the steps of imparting high velocity turbulent flow to a gas stream and directed against a heated object with said gas stream while the gas is moving at a velocity between about 40 and 90 feet per second, and maintaining the ambient gas temperature at approximately 125° F.

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