

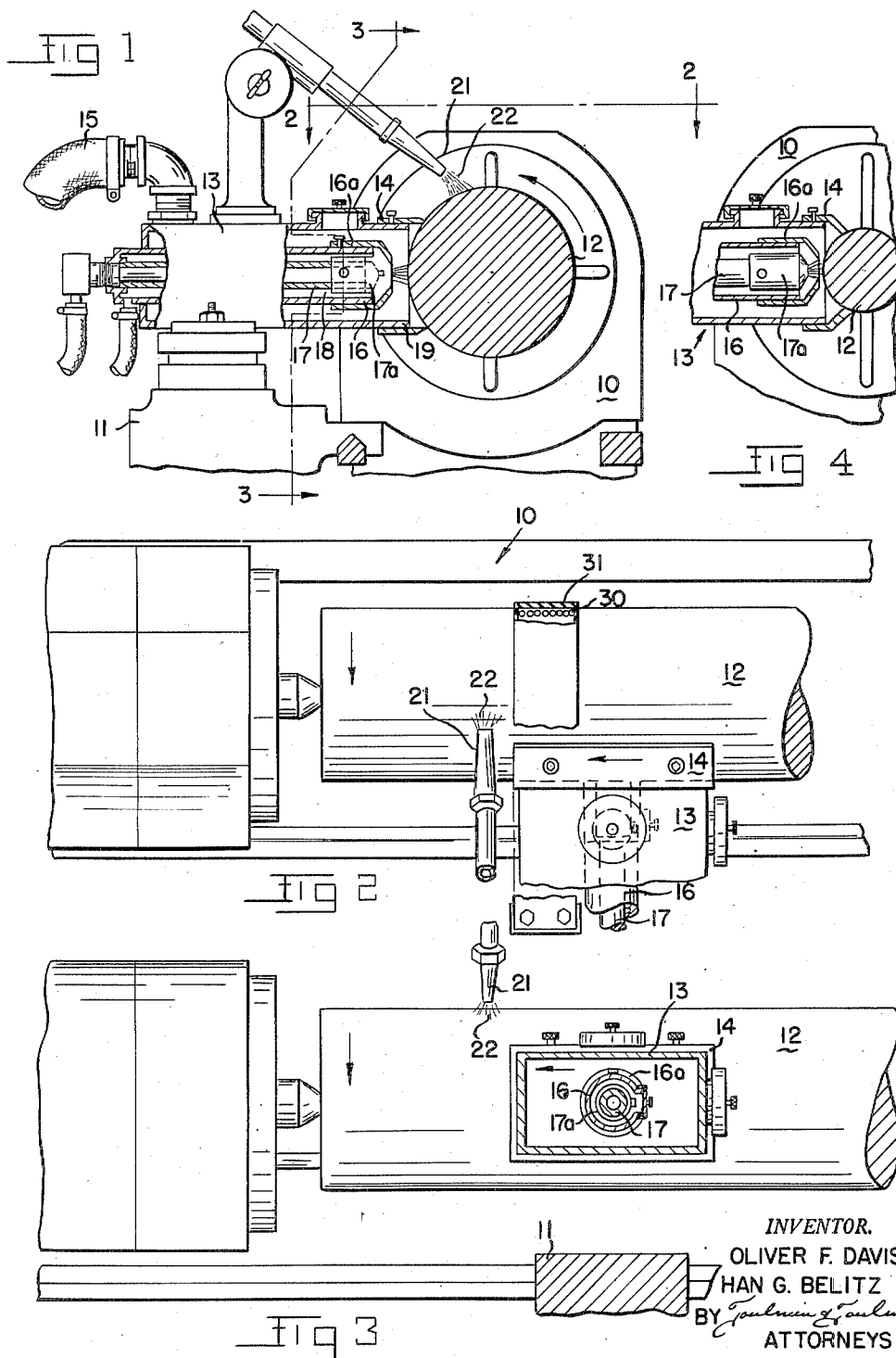
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H. G. BELITZ ET AL

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METHOD FOR GAS PLATING

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INVENTOR.

OLIVER F. DAVIS

HAN G. BELITZ

BY *Pauline J. Pauline*
ATTORNEYS

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METHOD FOR GAS PLATING

Hans G. Belitz and Oliver F. Davis, Dayton, Ohio, assignors to The Commonwealth Engineering Company of Ohio, Dayton, Ohio, a corporation of Ohio

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1 Claim. (Cl. 117—104)

This invention relates to the art of deposition of metals, and is a division of our co-pending application, Serial No. 94,804, filed May 23, 1949, now Patent No. 2,631,948. More particularly it relates to coating of metal bases. Still more particularly it relates to the plating of objects by the deposition of metal from readily decomposed volatile metal bearing compounds and apparatus for carrying out the process.

Depositing of thin film of metals, such as iron, nickel and cobalt, upon metal bases has been accomplished in the past by enclosing an object to be plated in a chamber sealed against entry of air.

The chamber was purged with carbon-dioxide and the object heated at a temperature at which volatile metal carbonyls decompose. Following this a metal carbonyl gas was carbureted into a stream of carbon-dioxide to form a dilute carbonyl medium which was metered into the chamber. Upon coming in contact with the hot object the carbonyl was decomposed and the metal component deposited.

This process has many disadvantages which limit its usefulness. While the chamber is filled with a mixture of inert gas and metal carbonyl vapors, plating only occurs when the carbonyl vapor contacts the hot object and general composition may take place, with the result that powdered metal accumulates in the bottom of the chamber.

Further, the deposition rate is very slow and the process requires hours to build up an appreciable depth of metal coating. In addition, the coatings are brittle and adhered poorly to the base metal.

In another process, utilizing quite similar equipment, the brittleness and poor adhesion has been largely overcome. In this process the metal deposition is carried out in two stages.

After a thin layer of metal is deposited the object is subjected to heat treatment at a temperature of between 500 and 800° F. to desorb the occluded gases. The object was then returned to the chamber and a second coating of the desired thickness over the first layer deposited thereon.

This process, while producing adhering metal coatings, is still a time consuming one. It also requires that the plating cycle be interrupted with consequent loss of materials due to purging the equipment to avoid formation of explosive mixtures of carbonyl gases with air.

Both of these processes, in addition to the disadvantages mentioned, require that the plating process be carried out in a closed chamber. This necessitates complicated inlet and egress apparatus if the operation is to be made continuous, or requires that the chambers be large and unwieldy if anything is to be handled other than small objects.

It is an object of this invention to overcome the limitations and disadvantages of the above described processes.

It is a further object of this invention to provide a process which eliminates the need for a complicated pressure chamber and its auxiliary equipment.

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A still further object of this invention is to provide a process wherein small areas can be plated either as initial coatings or for repair of damaged coatings.

A still further object of this invention is to provide a process which advantageously plates on rotatable objects of considerable thickness.

Still another object of this invention is to provide a process which lends itself to equipment arrangement capable of fast cooling whereby oxidation of plated coats is under direct control of the operator.

It is another object of this invention to provide a process in which the time for depositing any thickness of coating is markedly shorter than heretofore.

It is another object to provide a process which may be operated under either positive or negative pressure conditions.

It is a further object of this invention to provide a process wherein the decomposition gases are quickly removed from the plating area to eliminate contamination of the deposited metal film.

It is still a further object of this invention to provide a process for continuous and rapid deposition of bright metal coatings by directing the decomposable metal material to the metal surface.

It is a still further object of this invention to provide a simplified method and apparatus for depositing metal from a volatile metal compound by continuously decomposing the compound and conducting the gaseous product resultant from the decomposition away from contact with hot metal in order to avoid contamination and dulling of the bright deposit.

It is still another object of this invention to provide a process wherein the decomposable material is not brought up to a decomposition temperature before it is in the direct plating zone.

Other and more specific objects and advantages will be apparent to one skilled in the art as the following description proceeds:

The principal step of coating base material in accordance with this invention comprises causing a jet of decomposable metal compound vapors in a concentrated form to impinge upon an area to be plated. This jet is preferably brought to a temperature approaching the decomposition temperature before contacting with, for example, a rotating or moving base which is heated by suitable means in close proximity to the plating area.

The jet is arranged in cooperative relationship with a hood adapted to fit in close proximity to the object and designed to have a gas capacity capable of handling the many volumes of gas released by the decomposition of each molecule of volatile compound.

In this process the jet of vapor projected may be formed by mixing an inert gas with the vapors of the metal compound or by atomizing liquid metal compound with a blast of hot inert gas capable of converting liquid droplets to vapors and bringing the vapors to a temperature at which the vapors are on the verge of decomposing and needing only contact with hot metal to free the metal components.

The atmosphere in the vicinity of the surface to be coated is thus made up merely of fresh vapors propelled with force by the blast into the deposition zone and encountering mainly the hot gaseous decomposition products which are being removed, by example, as by suction or vacuum drawn on the area surrounding the plating surface.

Plating material is driven to the plating surface by a blast of inert gas. This inert gas is fed to the mixing point usually through an annular chamber surrounding a central feed pipe.

Carbon dioxide, helium, nitrogen, hydrogen, the gaseous product of controlled burning hydrocarbon gases

free of oxygen, and the like, have been utilized as the gas blast.

We prefer to utilize gas held under pressures of between 30 to 90 pounds per square inch for blasting, but the pressure range is subject to much wider variation, depending upon whether liquid or vapors are being sprayed in a limited area jet toward the plating surface.

Metals 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 carbonyl halogens, for example, osmium carbonyl 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 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.

Maintenance of the metal objects at temperatures in the general operating range is easily accomplished with numerous heating means, such as radiant heating, electrical resistance heating, induction heating, and the like.

The material to be decomposed is readily brought to the decomposition temperature by mixing with hot inert gas. Even a fine spray of liquid can be transformed from a liquid at a temperature of 100° F. to a vapor having a temperature of 325° F. in a fraction of a second, and the vapors being decomposed by contact with a heated surface having an even higher temperature.

Preparatory to coating base material the metal strip or object to be plated may be cleaned by employing the conventional methods used in the art, comprising electrochemically cleaning by moving the same through a bath of alkali or acid electrolyte, wherein the strip is made the cathode or anode. Pickling of the metal with hydrochloric, sulfuric or nitric acid, or a combination of acids may also be made as a part of the cleaning process, and the strip thoroughly rinsed or washed prior to introduction into the plating apparatus of this invention.

The invention will be more clearly understood from the following description of one embodiment of the apparatus wherein the object to be coated is a rotating shaft:

In the drawings:

Figure 1 is a sectional view diagrammatically illustrating a spray and exhaust apparatus;

Figure 2 is a view of the equipment of Figure 1 along line 2—2 and showing alternative or cooperative electrical heating means mounted within the hooded area;

Figure 3 is a view along the line 3—3; and

Figure 4 is a sectional view diagrammatically illustrating a hood capable of enclosing a greater proportion of the surface area of a circular object.

Referring to the drawings, there is shown a portion of a lathe 10 having a movable carriage 11 associated therewith, in which is mounted a metal shaft 12. The direction

of rotation of the shaft is shown by the arrow. Mounted on carriage 11 is a hood 13 having an adjustable head section 14 arranged as further illustrated in Figures 2 and 3. This hood is operatively connected with a source of vacuum or high capacity exhaust fan, not shown, through conduit 15.

Axially positioned within hood 13 is a pipe 16 which in turn has within it a feed pipe 17. Each of these pipes is provided with an adjustable end section as shown at 16a and 17a. An annular space 18 between pipes 16 and 17 provides a conduit for introduction of gas to the orifice area.

Pipe 17 serves to conduct vapor of the metal compound to an orifice 19 which directs the gas stream to the segmental surface area in alignment with the gas jet blanked by said hood 13.

Outside the hood 13 is mounted a nozzle 21 directing a flame 22 against the surface of the shaft 12. The flame is supplied with combustible gas from a source not shown.

Referring to Figure 2 there is shown a shaft 12, hood 13, vapor orifice 19 corresponding to their counterparts illustrated in Figures 1 and 2.

Mounted on the base 11 is an electrical resistance coil 30 through which the shaft 12 is free to rotate. Coil 30 is covered by rubber sheaving 31 to prevent metal from adhering thereto.

It will be noted that in this instance the heating equipment is mounted within the hooded section.

Conditions of operation with relation to specific plating operations carried out in the above described apparatus will be more fully set forth in the following examples:

Example I

An alloy steel shaft may be positioned in the lathe 10. The segmentally shaped hood 13 supported by the carriage may be lowered into close proximity to the shaft. Nickel carbonyl in the form of vapor may be fed through the orifice 19 at a rate of about ¼ pound of nickel carbonyl per minute. The carbonyl vapors may be driven to the surface of the shaft by a blast of carbon dioxide gas maintained under a pressure of 30 pounds per square inch. The shaft may be maintained at approximately 390° F. by the flame 22. The hood 13 may be maintained under a slight vacuum of approximately 2 inches of water in the exhaust conduit. At the above flow rate 50 square inches of shaft surface may be plated with a smooth continuous coating of nickel in less than one minute.

Example II

A wrought iron strip which is to be coated may be supported on a non-metallic conveyor, the strip may be moved in a straight path and pass under a hood which operates in the same manner as the hood of Example I.

This hood differs from the hood of Example I only in that it is shaped to fit closely to a flat object. The hood may be positioned in close proximity to the surface of the iron strip. When the metal has been heated to about 375° F. vapors of iron carbonyl flowing at the rate of 70 cubic feet per hour may be introduced through the nozzle within the hood and projected by the use of nitrogen gas.

The exhaust mechanism may be adjusted to maintain approximately 1 pound positive gas pressure in the plating vicinity.

With the nozzle adjusted to direct iron carbonyl vapors the full width of the iron strip, the strip may be continuously plated with a smooth iron deposit of relatively even thickness.

Example III

Copper disks may be supported on the conveyor of Example II and the same equipment utilized for the plating operation.

When the copper disks have been heated to approxi-

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mately 420° F. vapors of antimony hydride may be introduced through the nozzle within the hood and projected by the use of hydrogen gas supplied under a pressure of 60 pounds per square inch.

The exhaust mechanism may be adjusted to maintain approximately 1 to 2 inches of water negative pressure in the outlet section of the hood.

With a flow rate of vapors of approximately 100 cubic feet per hour of antimony hydride, small copper disks may be plated with smooth coating of antimony in the matter of seconds.

Example IV

In this instance a steel shaft is gas plated with molybdenum metal. The method comprises rotating the shaft while providing a protected surface area of the shaft as in Example I and where inert gas atmosphere prevents ingress of air to the plating area, impinging upon the surface area a vapor stream consisting of hydrogen and molybdenum carbonyl, heating the shaft locally to a temperature in the range of 350° F. to 425° F., and removing the hydrogen and carbon monoxide waste products under conditions to maintain mixing of air into the exhaust stream to a minimum.

It will be understood that while there have been given 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 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 this disclosure and the scope of the appended claim.

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We claim:

The method of plating steel shafts with molybdenum metal deposited from a heat-decomposable metal vapor which comprises the steps of restricting the free access of air to a limited surface area of the shaft to be plated, establishing a source of heat-decomposable molybdenum metal vapor in the form of a jet under pressure onto said limited surface area to be plated, said metal vapor consisting of hydrogen and molybdenum carbonyl, and subjecting said area to localized heating to raise its temperature thereof to between about 350° F. and 425° F. to cause said impinging metal vapor to decompose and deposit molybdenum metal onto said limited surface area, and removing the decomposition products under conditions to maintain mixing of air into the exhaust stream to a minimum, said removal taking place in a direction opposed to that of said given direction.

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