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APPARATUS AND PROCESS FOR PRODUCING COATINGS ON METALS

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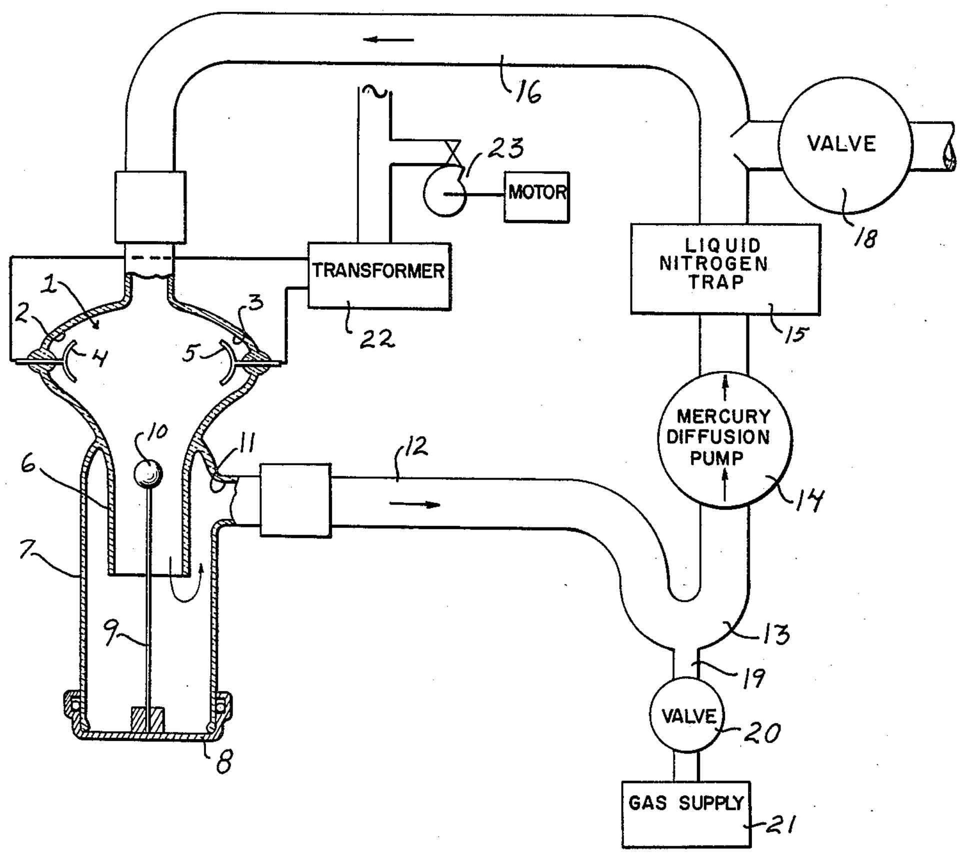

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APPARATUS AND PROCESS FOR PRODUCING COATINGS ON METALS

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This invention relates to apparatus and process for producing coatings on metals and included in the objects of this invention are:

First, to provide an apparatus and process for producing coatings on metals wherein nitrides, oxides, hydrides and carbides or the like may be deposited on and bonded to a cold metal surface without injuriously heating the metal, for purposes of corrosion prevention, hardening or for other purposes, or may be prepared in the pure state if desired.

Second, to provide an apparatus and process for coating metals wherein the metal to be coated is placed in a stream of a gas under a sufficiently reduced pressure to sustain a glow discharge and the gas continuously circulated while a glow discharge is maintained in the region.

Third, to provide an apparatus and process for coating metal wherein the atomization of the gases may be pulsed in accordance with a predetermined schedule to minimize heating of the target and also minimizing diffusion of the reacting gases, thereby minimizing such deleterious effects as embrittlement.

Fourth, to provide an apparatus and process of coating metal wherein the atoms of gas which become the coating are swept toward the target; that is, on to the metal to be coated by the physical circulation of the gases as distinguished from the impelling of the atoms of gas by electrostatic attraction; this results in a substantial reduction in the energy requirements to effect a given coating.

With the above and other objects in view as may appear hereinafter, the accompanying drawing is a diagrammatical view of apparatus to perform the invention.

A glow discharge tube 1 is constructed of glass or other suitable material and is provided with two opposing cavities 2 and 3 in which are mounted electrodes 4 and 5, or other conventional means for effecting a glow discharge, such as a coil surrounding the glow discharge tube and connected to a source of high frequency current. The tube 1 converges downwardly into a neck 6 which projects into a chamber 7. The upper end of the chamber is joined to the neck above the lower end of the neck. The lower end of the chamber which is generally cylindrical is provided with a removable base member 8 which may be provided with a rim which overlaps the lower end of the chamber and which is provided with an O-ring or other suitable seal means. Supported in the bottom member is an upright stem or target holder 9 which projects into the neck 6 and at the upper end of which is mounted a specimen or target 10 which is intended to be coated. The upper end of the chamber 7 above the lower end of the neck 6 is provided with a lateral outlet 11 which is connected to a circulating tube 12. The circulating tube includes a trap 13 then continues to join to the inlet end of a pump 14. The discharge end of the pump 14 communicates with a trap 15 capable of trapping mercury vapor efficiently but not cold enough to freeze out the reacting gas. The outlet end of the trap 15 communicates with a return line 16 which extends to and communicates with the upper end of the glow discharge tube 1. Between the nitrogen trap 15 and a glow discharge tube 1, the return line 16 communicates with an exhaust line 17 which leads to a roughing pump, not shown, and in which is mounted a valve 18.

The trap 13 is connected with an inlet tube 19 controlled

by a valve 20 and which is connected to a gas supply 21, the gas supply containing the gas which is utilized to effect a coating of the specimen or target. The electrodes 4 and 5 may be connected to a constant source of high voltage energy. However, it is preferred to connect the electrodes 4 and 5 to a transformer 22, the input of which is controlled by a pulsing means 23, as, for example, a switch and a cam for closing the switch periodically and thus energizing the electrodes periodically.

The specimen or target which by way of example may be an article of titanium or any other metal to be treated is positioned in the neck 6 and supported there by the holder 9. The system is evacuated and then filled with nitrogen to a partial pressure in the range of 8 mm. Hg and a reacting gas at a pressure in the order of 0.5 to 3 mm. Hg. The pump 14, which is a diffusion pump, is started causing circulation in the direction indicated.

The transformer 22 is operated to deliver voltages in the range of 15,000 volts for a few seconds. This produces a greater discharge between the electrodes but little or no glow in the region of the sample. The transformer is caused to operate intermittently after a short interval such as five minutes. The glow virtually disappears indicating that the nitrogen has reacted. At the same time a faint golden color appears on the sample indicating that some nitriding of the surface has occurred. If it is desired to provide a continuous system, nitrogen may be supplied through the valve 20 at approximately the rate at which the nitrogen is reacting so as to maintain a substantially constant vapor pressure.

The process is suited for coating precision parts on which further grinding or machining is not tolerated. While nitrogen is mentioned, other substances may be used including hydrocarbons such as the aliphatic types, as, for example, methane, ethane, etc. However, any gas which will yield free radicals or atoms in a glow discharge may be used. If the coating produced by such gases is desirable; for example, the halogen gases, chlorine and fluorine, may be used. In addition other materials which become gases at operating pressure or temperatures may be used. Among these are such substances as carbon tetrachloride.

The reduced pressures should be such that the gas has sufficient mean free path to serve its purpose before recombination and the range varies from 0.5 to 1.0 mm. Hg for hydrogen to 5 mm. for halogen gases. If a diluent gas is used, it should be an inert gas such as argon or neon. The pressure range of the diluents should be between 10 and 30 mm. Hg if the tube is approximately 30 mm. in diameter. For larger tubes the pressure should be lowered.

It should be observed one electron volt is equivalent to 23,200 calories per mole. From the kinetic theory of gases, the energy (and consequently the temperature) is equivalent to about 7700° K. per electron volt. For example, hydrogen (103,000 cal per mole for dissociation energy), the temperature of the hydrogen atom will be about 38,000° K. (5 electron volts). For chlorine this would be about 20,000° K. Nitrogen, to name one of the more common gases, has a high dissociation energy corresponding to about 70,000° K.

With regard to the pulsing of the transformer, the frequency is such as to avoid overheating the specimen, for if pulsing were not carried out a continuous stream of "hot" atoms would tend to melt the specimen. The coating thickness ranges from a few Angstroms to the order of a few ten thousandths of an inch.

Some inter molecular penetration or diffusion of the coating may occur, but not to any substantial depth without excessively heating the specimen. As it is desirable to avoid high temperatures this would defeat the purpose;

namely, to effect a substantial cold coating without distorting or changing the crystal structure of the specimen.

Having thus described certain embodiments and applications of my invention, I do not desire to be limited thereto, but intend to claim all novelty inherent in the appended claims.

I claim:

1. A process for coating metals, characterized by: circulating repeatedly a coating gas and a diluent gas within a closed loop system at partial pressures capable of sustaining a glow discharge; producing a glow discharge in a selected region of said system; positioning a specimen to be coated contiguous to and downstream of said glow discharge to collect a deposite of a portion of said coating gas; said substance carried downstream of said glow discharge region, the remaining portion of said coating gas recirculating through said system for subsequent passage through said glow discharge and to the area around said specimen, and adding additional coating gas into the system to maintain the partial pressure at a predetermined value capable of sustaining a glow discharge.

2. A process as set forth in claim 1 wherein: said glow discharge is maintained at a substantially steady state.

3. A process as set forth in claim 1 wherein: said glow discharge is pulsed to provide periodic glow discharge.

4. An apparatus for coating metals, comprising: a circulating conduit forming a closed loop and adapted to contain a coating gas at a pressure capable of sustaining a glow discharge; means for circulating said coating gas

repeatedly through said conduit; means defining a glow discharge chamber and a contiguous specimen chamber interposed in said conduit said specimen chamber being downstream of said glow discharge chamber; means for producing a glow discharge in said glow discharge chamber; means for introducing additional coating gas into said system to maintain said pressure at a level capable of sustaining said glow discharge; and means for positioning a specimen in said specimen chamber in the path of said coating gas for reaction of said gas with the surface of said specimen.

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