DEPOSITION OF POLYMERIC COATINGS UTILIZING ELECTRICAL EXCITATION

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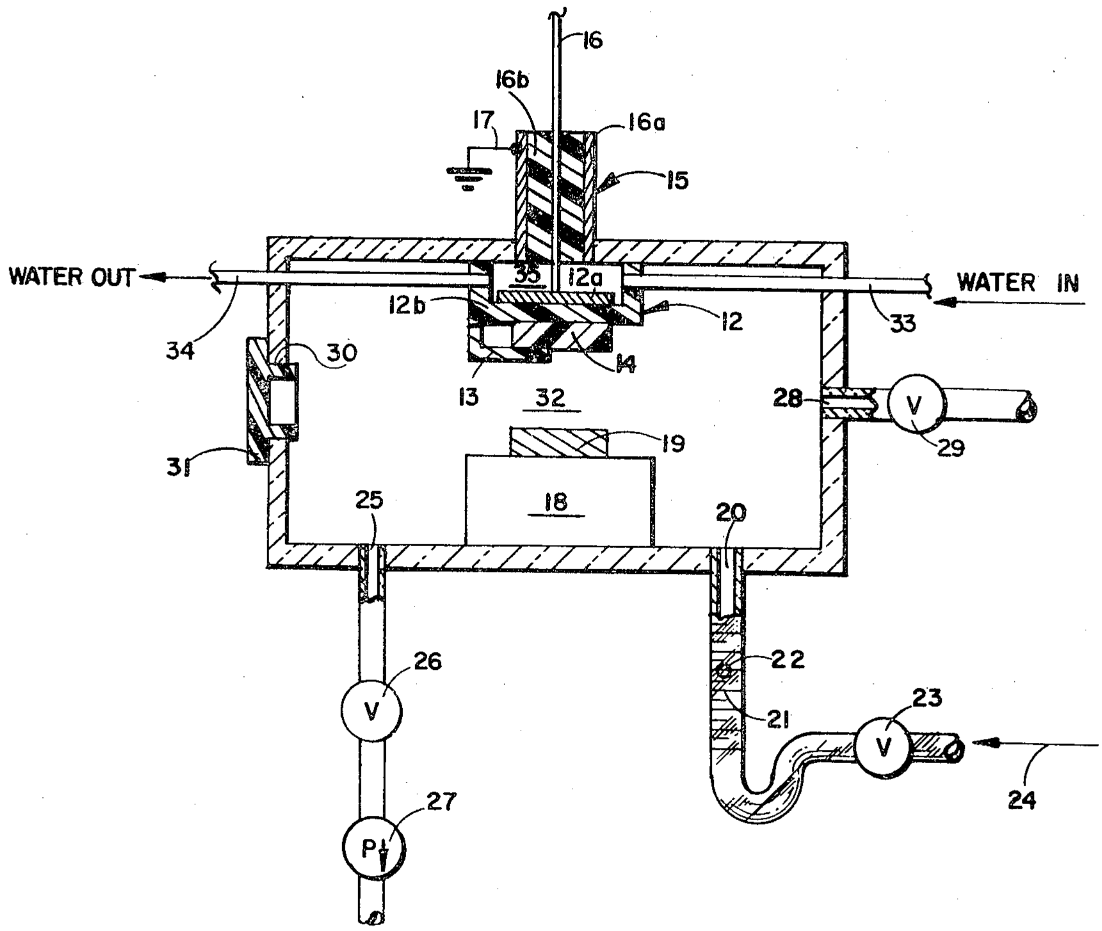

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DEPOSITION OF POLYMERIC COATINGS UTILIZING ELECTRICAL EXCITATION

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ABSTRACT OF THE DISCLOSURE

An electric deposition process providing a glow discharge by virtue of electrical energy fields provided internal to a deposition chamber enables with one step to provide a protective coating of a surface of a substrate material by polymerizing a dimer solid starting material. A carrier gas such as argon, xenon or krypton is used in the deposition chamber for the energy field to act upon it, provide the glow and cause the glowing gas to volatilize particles of the starting material and be deposited as the protective coating on the substrate.

BACKGROUND OF THE INVENTION

The invention is in the field of a process and an apparatus therefor for depositing a film on a substrate by the use of electric or glow discharge principle. Certain prior art most closely related is hereinafter described as follows:

U.S. Pat. No. 3,462,335 to Hansen utilizes a glow discharge apparatus only for roughening or preparing a surface of one of two materials to be adhesively bonded to each other. However, in this process only one of the two materials is subjected to the glow discharge occurring by introduction of the apparatus of a helium and hydrogen gas mixture. It is then removed from the apparatus and a bonding material is applied to the prepared surface and the other material attached to the material which has the bonding material applied thereto.

U.S. Pat. No. 3,475,307 to Knox utilizes a glow discharge apparatus in which is situated a material to be coated. However, the coating is accomplished by introducing a gaseous hydrocarbon monomer therein. Also the glow discharge apparatus utilizes electrodes therein with a high voltage applied thereto. Additionally, no carrier gas is used for conducting the monomer and for maintaining the glow discharge.

U.S. Pat. No. 3,310,424 to Wehner utilizes a glow discharge apparatus for providing a coating on a substrate surface. However, the coating starting material is a liquid, and the liquid used is a silicone. Also, the apparatus uses electrodes therein to effect a discharge therebetween, and is dependent upon a starting igniter in a mercury pool within the apparatus.

U.S. Pat. No. 3,457,156 to Fisher utilizes a glow discharge apparatus for providing a polyacetylene coating on a substrate. The apparatus has electrodes therein and an acetylene gaseous starting material is injected into the apparatus to be ignited by the apparatus. No carrier gas is used to facilitate glow discharge activity.

U.S. Pat. No. 3,406,040 to Da Silva utilizes an apparatus having an electron beam structure therein. The starting material is a non-volatile evaporant which is evaporated by heating in a stream and subjected to the electron beam which causes the stream of evaporated material to be formed as a coating on the substrate positioned in the apparatus.

U. S. Pat. No. 3,449,154 to Katz utilizes a glow discharge apparatus having electrodes therein to create the electrical discharge therebetween, for providing a coating on a substrate. However, the starting material is a liquid which is vaporized into a gas for vapor deposition on the surface of the substrate. The starting materials are lactams and lactones in liquid form.

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U.S. Pat. No. 3,318,790 to Carbajal shows an electric discharge apparatus embodying the principle of the vacuum tube. An electric arc is created between a cathode and an anode, and the perforated cathode permits electrons, or part of the arc to pass therethrough and impinge on the surface of the substrate in the apparatus. Simultaneously, a starting material in the form of an organic gaseous source material is injected into the apparatus upon which the arc portions passing through the cathode act for deposition of a film on the substrate.

U.S. Pat. No. 3,472,679 to Ing shows an apparatus used for deposition of starting material as a film on a substrate. The starting material is an unpolymerized solid consisting of selenium, arsenic, tellurium, antimony, bismuth, thallium, sulphur and mixtures thereof. Also usable are halogens such as chlorine and iodine plasmas in connection with the starting materials. A plasma is created by ignition of a carrier gas in the vicinity of the substrate, and the starting material is vaporized depositing as a film on the substrate.

U.S. Pat. No. 3,069,283 to Coleman provides in part a method utilizing an evacuated apparatus and an electric discharge principle for providing a polymerized coating on a substrate. However, there, the starting material used to provide the coating is a gaseous monomer supplied by an external tank into the apparatus.

U.S. Pat. No. 3,068,510 to Coleman provides in part a method utilizing an evacuated apparatus and means for producing electric discharge in the apparatus to provide a polymerized coating on a substrate having an electrically conductive surface. Power is provided for polymerization of the gaseous material, which is contained in an external tank. The substrate to be coated being conductive is part of the electrical circuit to which the power source is connected. The gaseous material introduced into the apparatus provides the medium through which an electric discharge occurs, depositing the gaseous starting material previously introduced into the apparatus on the electrically conductive substrate.

INVENTION SUMMARY

Briefly, according to the invention, an electric deposition process is utilized to provide an organic coating on a substrate material. The process uses a dimer starting material positioned, with respect to a substrate material which surfaces are desired to be coated, in an evacuated chamber into which a carrier gas of argon, krypton or xenon, or mixtures thereof are injected into the chamber. The chamber is provided with means for electrically exciting the carrier gas, and setting up a glow therein of the carrier gas. The starting material is bombarded with the electrically excited carrier gas which volatilizes portions of the dimer starting material which passes into the glow zone. This action causes deposition of the volatilized material on the surface of the substrate material forming a polymerized coating of the dimer starting material on such substrate surface.

An object is to overcome the disadvantages of the prior art, which is mainly possible by utilization of radio frequency energy creating a glow of the inert carrier gas used in the process acting on the solid dimer starting material, and not possible by any other process.

BRIEF DESCRIPTION OF DRAWING

The figure is a schematic representation of a processing chamber energizable with radio frequency energy, evacuated, and into which a carrier gas is injected for maintaining a glow discharge to act upon the dimer solid material in the chamber and to provide the deposition of a polymerized protective layer on the substrate prepositioned in the chamber.

EXEMPLARY EMBODIMENT

Referring to the figure, chamber **11** made of glass or other insulating material is provided to practice the process.

Radio frequency radiator **12** generally having external surfaces made of, or covered with electrically insulating material such as a ceramic, is attached to the upper surface of the inner chamber wall. Radiator **12** has a holder **13** made of suitable material such as a ceramic for holding in place the dimer solid material **14**.

Examples of dimer solid materials are of the di-para-xylene, di-chloro-para-xylylene and di-chloro-para-xylylene but are not limited necessarily thereto.

One advantage of placing material **14** near radiator **12** is to enable greater efficiency in transmitting or saturating the material with radio frequency energy. The carrier gas injected in chamber **11** is excited by the radio frequency energy emanating from radiator **12**, sets up a field in the chamber generally resulting in a glow due to excitation of the gas between the radiator and the substrate material to be hereinafter described.

To achieve the radio frequency energy field, one end of a coaxial cable **15** is connected to the input of radiator member **12**, by connecting center conductor **16** to metallic radiator element **12*a***, insulation holder **12*b*** electrically insulates element **12*a*** from the starting material **14**, and outer conductor shield **16*a*** which is part of cable **15** is grounded as at **17**. The other end of center conductor **16** and outer conductor shield **16*a***, which is grounded at **17**, is connected to the radio frequency power source (not shown). Insulator **16*b*** electrically insulates shield center conductor **16** from shield **16*a***.

The chamber is provided internally with a pedestal **18** on which the substrate material **19** desired to be coated is positioned.

The chamber is provided with a port at **20** to which is attached one end of flowmeter **21**. The flowmeter is calibrated to measure flow rates of carrier gas by means of a floating ball **22** internally thereto. The higher the floating ball rises by virtue of carrier gas passage through the flowmeter, the greater the flow rate of the carrier gas.

Flow regulating valve **23** is attached to the other end of flowmeter **21** for controlling the flow rate of carrier gases injected therein as diagrammatically indicated by arrow **24**. Carrier gases normally used in this process may include argon, krypton and/or xenon, and are injected at the rate of 300 cc./min. during the process.

Port **25** is provided in the chamber for attaching one end of a vacuum control valve **26** for regulating speed of evacuation of the chamber or for completely shutting off the evacuation of the chamber.

Pump **27** is attached to the other end of valve **26**, and is used to maintain the desired amount of vacuum. Typical vacuum range has between 0.01–0.03 millimeter of mercury.

Port **28** is provided in the chamber for attaching bleed valve **29** thereto which is closed during deposition cycle of the process and is opened at completion of the deposition cycle to release the vacuum in the chamber.

Port **30** is provided in the chamber for both loading the the starting material and the substrate into the chamber at the beginning of the process, and is also used to remove the substrate material at the end of the process and also any unused portion of the starting material. Door **31** is a vacuum tight cover preventing leakage during the process and is opened after release of vacuum. Naturally, it is opened in order to load the chamber with the starting and substrate materials.

Water inlet is provided at input of pipe **33** for circulating water over the surface element **12*a*** so as to cool same since about 300 watts of electrical power will be supplied from the RF power supply, and pipe **34** is provided for dumping the water after circulating in cavity **35** of radiator member **12** between the glass chamber **11**, member **12*a*** and insulation holder **12*b***.

Typical experimental examples including parameters used, successfully accomplishing the objectives of this process, are given in the following table, but are not limited to the examples shown in such table:

Dimer solid starting material	Temperatures, °C. Radiator	Substrate	Pressure [1]	Inert carrier gas used
Di-para-xylylene	50	50	.01	Argon.
Di-chloro-para-xylylene	75	75	.02	Xenon.
Di-dichloro-para-xylylene	65	25	.03	Krypton.

[1] In millimeter of mercury to maintain glow of carrier gas.

Hence, in the experimental examples stated above, with substrate and starting materials in place, door **31** closed, valve **29** closed, valve **26** open, pump **27** in operation evacuating the chamber to the required pressure as given in the table in order to maintain the glow, power supplied to terminals **16–17** and water is supplied to pipe **33**, valve **23** is opened to allow carrier gas **24** to be injected in the chamber and adjusted to provide maximum glow of the RF excited carrier gas.

The excited carrier gas, now glowing, bombards the dimer starting material **14**, knocking out or volatilizing the molecules of material **14** into glow zone **32** and results in a polymerized coating on such surfaces of substrate which are exposed.

I claim:

1. An electric deposition process for providing an organic coating on a substrate material, said process utilizing an organic dimer starting material preselectably positioned with respect to said substrate material in an evacuated chamber into which an inert carrier gas selected from the group of at least one gas consisting of argon, krypton and xenon injected into the chamber, said chamber being provided with means for electrically exciting said carrier gas therein, the improvement comprising:
   bombarding said dimer starting material with the excited carrier gas thereby volatilizing portions of said starting dimer material which pass into the glow zone of the chamber thereby depositing the volatilized material on a surface of said substrate material in the form of a polymerized coating of said starting material, the organic starting material being at least one dimer selected from the class consisting of di-para-xylylene, di-chloro-para-xylylene and di-dichloro-para-xylylene.

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