

3 Sheets-Sheet 1



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KIYOSHI INOUE
METHOD OF AND APPARATUS FOR THE ELECTRIC
SPRAY-COATING OF SUBSTRATES

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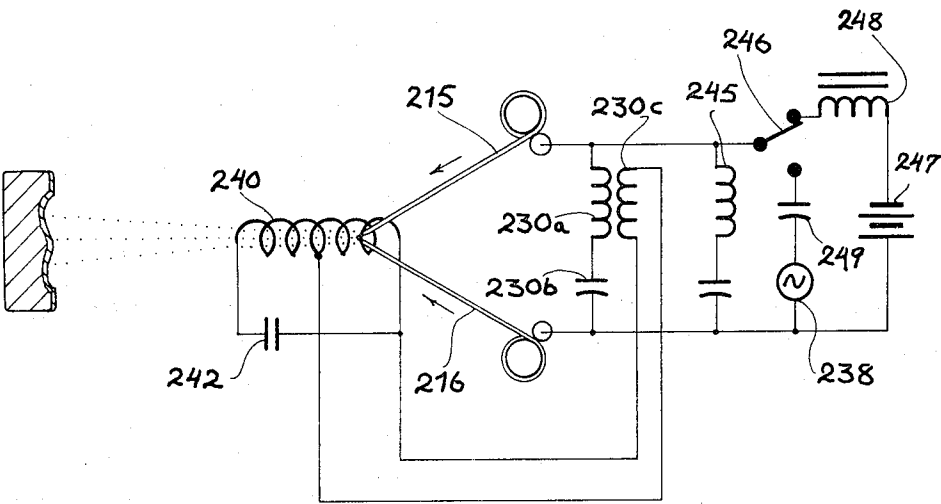


Fig. 3

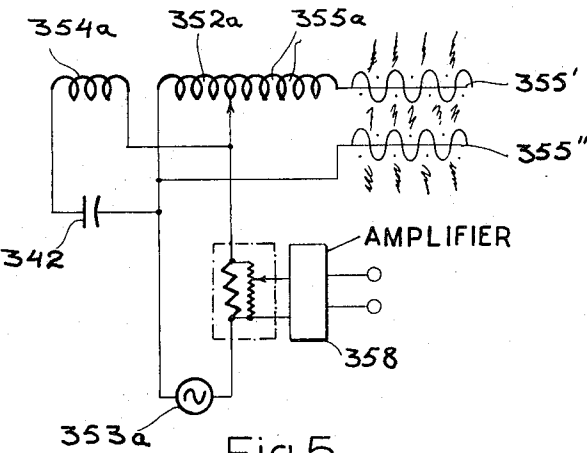


Fig. 5

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METHOD OF AND APPARATUS FOR THE ELECTRIC SPRAY-COATING OF SUBSTRATES
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ABSTRACT OF THE DISCLOSURE

Method and apparatus for the electric spray deposition of particles of a solid substance upon a substrate wherein the particles are formed by heating a rod of this first substance and the particles are entrained in a gaseous stream in the direction of the substrate, electrodes along the path generate a discharge for ionizing the gaseous stream while a magnetic coil downstream thereof provides a focusing or shaping effect. A signal is electromagnetically derived from a coil downstream of the ionization for regulating the feed of the solid body, the rate of ionization thereof, the flow of the gas stream and the focusing current.

My present invention relates to metal deposition and, more particularly, to improvements in sputtering, spraying and other methods of coating a substrate with a metallic or metalloid layer.

In general, metal coating of substrates has been carried out in several ways depending both upon the metal to be applied and the nature of the substrate. It is well known, for example, that iron and steel can be coated with zinc or tin by dipping the substrate into a molten bath of these metals and that similar metals and others such as copper, chromium and nickel can be applied to conductive substrates by electrolytic deposition. Nonconductive materials have been coated hitherto with gold, silver and metal salts by suspending or dissolving these salts in various solvents which then were evaporated to leave the desired coating or in vehicles decomposable at elevated temperatures. The latter technique is particularly effective for the overlaying of metal on glass, porcelain and ceramics since the metal can then be baked or fused into the substrate. In addition to these methods, it has become the practice of late to produce relatively thin metal coatings in, for example, the manufacture of so-called "metallized" foils, to vacuum deposit a metal upon a metallic or nonmetallic substrate. In this system, the metal is vaporized at reduced pressure and condensed upon the substrate which is relatively cool. Aluminized polyester films can be made in this manner.

In general, all of these purposes have been found satisfactory for some purposes but unsatisfactory for others. More specifically, it has been found to be difficult to provide uniform metallic coatings of greater than monomolecular thickness or on this order upon substrates with any of the aforescribed processes. Thus, it has been necessary to resort to sputtering or spark erosion of a metallic body with simultaneous deposition of the eroded material upon the substrate. In known sputtering techniques, a spark is developed between electrodes of the metal to be deposited closely adjacent the surface to be coated. Hot particles of the metal adhere readily to the relatively cool surface just as vacuum- or vapor-deposited metals bond to a substrate.

While a sputtered coating is more or less uniform, it has been found to be exceedingly difficult to direct the coating operation (i.e. to localize it) and to adjust the area of sputtering in accordance with particular needs. This difficulty arises from the fact that earlier sputtering

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methods required a certain minimum distance between the electrodes and the substrate to insure proper development of the metal sputtering stream but with a maximum distance determined by the density of this stream, which is relatively ill defined.

It is an object of the present invention to provide a method and apparatus for metal coating substrates whereby the aforementioned disadvantages can be obviated.

Another object of the invention is to provide a method of spray depositing a metal upon substrates whereby the deposit can be localized and confined to relatively small areas without the need for expensive and complicated apparatus.

A further object of the invention is to provide, in an apparatus for spray depositing a metal, improved means for controlling the spray.

These objects and others which will become apparent hereinafter are attained, in accordance with the present invention, by providing means for spray depositing a metal upon a substrate which includes a source of metal particles, preferably in a submicroscopic state, in the presence of an ionizable carrier fluid, electromagnetic means being employed to concentrate or control this carrier. I have discovered that even when such electromagnetic means are dispensed with, the ionizable carrier, if composed of a relatively electronegative material (i.e. a substance with an affinity for positively charged particles), forms a shell about the stream of metallic particles in some manner concentrating this stream and confining it to a relatively small area. The use of a carrier fluid with forced draft imparts a general drift to the metallic particles which is stronger than that normally noted in the environs of a sputtering device and insures a rapid transfer of the metal particles to the substrate disposed in the fluid path. In fact, it is another feature of the present invention to provide means for controlling at least one parameter of this fluid to regulate the intensity, velocity and/or shape of the metal-particle stream directed against the substrate. It is possible, by increasing the velocity of the gas stream, to increase the kinetic energy of the metal particles and thus improve the bond formed thereby upon contact with the substrate. Similarly, if a denser coating of the metal is desired, the volume rate of flow of the gas stream can be increased so that a more effective drift of metal particles away from the electrodes takes place. It is also contemplated, in accordance with the present invention, to dispense with the ionic fluid and to employ a non-ionized gas as the carrier fluid. In the latter case, the gas can be joined with the metal-particle stream forwardly of the electrodes. It is envisaged, in accordance with the instant invention, to provide automatic control means for adjusting a parameter of the gas flow in accordance with the rate of metal erosion or with the desired intensity and areal coverage. Similar means can be employed for correlating both metal erosion and gas flow as well as the degree of gas ionization.

While it is preferred to produce the metal particles by spark or arc discharge, it should be noted that other means can be utilized within the contemplation of the present invention. More particularly, a metal to be deposited upon a substrate can be heated, according to a further aspect of the invention, preferably inductively although other conventional means such as a gas torch may be employed, and carried by the gas stream onto the substrate. According to another facet of this particular phase of the invention, a metallic body is inductively heated and servo-control means are provided to correlate the rates of heating, gas flow and the metal supply. As previously mentioned, it is desirable to provide electromagnetic means for confining the beam of metallic particles. When spark erosion is employed, both the carrier gas and a substantial proportion of the metal particles are

found to be in an ionic state and readily susceptible to control by electromagnetic means. Similarly, vaporization or erosion of a metallic body by induction or other heating methods also results in a high proportion of ionic particles. This proportion can be increased substantially when spark discharge and/or an actuation frequency on the order of the ionization resonance frequency of the metal as described in my copending application Ser. No. 262,466 filed Mar. 4, 1963, is used. In general, the electromagnetic control means will include one or more coils adapted to provide a magnetic field in the region of the exit aperture to disperse or concentrate the particle beam. The effect of this action can be similar to that employed in a process described in my copending application Ser. No. 273,480, filed Apr. 16, 1963 and issued Oct. 19, 1965 as Patent No. 3,212,311, for the shaping of metallic bodies. The spark or arc discharge producing the ionized gas particles can be independent of or correlated with the discharge producing the metallic particles or the other heating means for this purpose and may be effected upstream from the latter in the direction of gas flow. A corona discharge, induced by the magnetic field of the control coils in an energizing coil, can be effected upstream of the region of metal-particle formation for producing ionized particles in the carrier-fluid stream.

Still a further feature of the instant invention resides in the use of the stream of ionized particles as a conductive stream of moving electrical charge to induce a focusing magnetic field capable of confining the beam of metallic particles or produce a control signal. Consequently, the electromagnetic control means can include a coil extending axially along the particle stream and preferably surrounding it, at least one portion of this coil being bridged by capacitive means to form with the remainder of the coil a parametric oscillator for energizing the entire coil in step or out of step with an electromagnetic control current. Advantageously, the capacitive means bridges the entire coil while a relatively high-frequency alternating-current is supplied to a portion thereof from a conventional A-C generator. While it is desirable that the metal particles be ionized for the present purpose, it should be noted that it is possible, according to the invention, merely to ionize the carrier gas and nevertheless obtain, in effect, an inductive feedback of the control signal to the valves, electrode-feed or particle supply means. When a spark-discharge generation of the metallic particles is dispensed with, it is contemplated, according to the invention, to use high-frequency inductive heating to decompose the metallic rod. Advantageously, this rod is of a magnetically permeable material capable of inducing an electric current in an auxiliary coil, axially aligned with the main or inductive heating coil to produce a focusing field therein substantially at the region of decomposition of the rod. The magnetic field and the focusing coil may be so dimensioned with respect to the charged particles passing therethrough that a defocusing or broadcast spray of the particles results. Additionally, one or another of the heating and particle-directing coils can be energized uni- or bidirectionally by inductive pick-up by virtue of the mechanical movement of the conductive rod.

An apparatus of the aforescribed type can be provided with conduit means via which a carrier gas is supplied to the region of decomposition of the metallic body, such conduit means having incorporated therein flow-control devices for regulating fluid flow in step with the rate of decomposition of the metallic rod. It is also possible to provide control means for displacement of the rod in accordance with the rate of its decomposition so as to maintain a constant spark gap if high-energy discharges are used for the decomposition or a constant location of the region of decomposition when inductive heating is used. Thus, it is another feature of the present invention to provide servo-control means coupled with the electromagnetic coils surrounding the rod or particle

beam for adjusting a flow-control means for the carrier-fluid and/or the feed means for the rod. In a modification of this technique, the servo-control derives a signal whose amplitude is a function of the magnitude of the inductive-heating effect. It is also contemplated to employ, for this purpose, a device capable of responding to increases and decreases in the discharge current in cases where spark discharge is used to ionize the particles.

The above and other objects, features and advantages of the present invention will become more readily apparent from the following description, reference being made to the appended drawing in which:

FIG. 1 is an axial cross-sectional view diagrammatically illustrating a device for the spray deposition of metals in accordance with the present invention, showing the control means therefor;

FIG. 2 is a very similar to FIG. 1 illustrating a modified circuit arrangement;

FIG. 3 is a circuit diagram of yet another apparatus of this general type;

FIG. 4 is a diagrammatic view of a system illustrating a modification wherein spark discharge is not employed; and

FIG. 5 is a diagram of a control system according to another embodiment of the invention.

In FIG. 1 I show a device for coating a mold or die 10 with a layer 11 of ferrochrome or the like. This process renders the die suitable for use with high-temperature injection-molding and die-casting machines even if the die body 10 is composed of a relatively soft metal (e.g. brass). It should be understood, however, that the apparatus described hereinbelow can also be used for the erosion of metal bodies by bombardment with charged particles with a proper selection of the impinging particles. The device comprises a nozzle 12 into which two inclined insulating sleeves 13, 14 are inserted, these sleeves forming guide bushings for a pair of metal rods 15, 16 passing therethrough. The rods are inclined forwardly in the nozzle and approach each other substantially along its axis and are unwound from respective supply reels 17, 18 by motors 19, 20, respectively. These motors drive respective feed rollers 21, 22 which bear against the rods 15, 16 and serve to provide the electrical connections therefor. The tubular nozzle 12 has an inlet 23 through which a carrier gas (e.g. air) may be induced by a Venturi effect. This effect is produced by an outer nozzle 24 surrounding the inner nozzle and forming therewith channels 25 inclined forwardly toward the axis. Intermediate these channels, there are provided angularly spaced ribs 26 by which the outer nozzle 24 is secured to the inner member 12. Conduit means, schematically represented by the dot-dash lines 27, are connected with the channels 25 and provided with a flow-control device such as the valve 28 communicating between these channels and a source of driving pressure, e.g. the pump 29. While it is desirable to control fluid flow by means of the valve 28, it should be noted that it is also possible to accomplish a similar effect by varying the rate of operation of the pump 29.

The rods 15, 16 which form an arc gap in the region of the axis of the nozzle, are energized by the secondary winding 30 of a transformer 31 whose primary winding 32 is bridged across an alternating-current source 33, such as a high-frequency generator. A current transformer 34 is connected in series with the rods 15, 16 and develops a signal indicative of the current flow through the arc and, consequently, the rate of decomposition of the rod material by this arc. The current transformer 34 supplies the signal, via a rectifier 35, to one winding of a servomotor 36 mechanically coupled with the valve member of valve 28. The other winding of the two-pole servomotor is energized directly by the secondary winding 30 through a rectifier 37. Reference potentials can be tapped off a high-ohmic resistor 38, connected across a battery 39 so that the desired flow rate can be set. When it is

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desired to spray-deposit ferrochrome upon the substrate 10, the rods 15, 16 both of which may be composed of a ferrochrome alloy or one of which may contain a substantial proportion of chromium while the other contains a substantial proportion of iron, are gradually brought together until a spark discharge is induced across their gap by the high-frequency source 30-32. The resulting arc decomposes the electrodes while a gas stream is forced through channels 25 to entrain the resulting particles in the direction of the substrate 10. Motors 19 and 20, which can operate at identical speed when both electrodes are composed of the same material or at different speeds in proportion to the rate of erosion of each electrode 15, 16 to maintain the gap substantially at the axis of the nozzle 12, can run at a constant rate or at a rate determined by the spray beam as will be apparent hereinafter. Carrier gas is induced through the interior of nozzle 12 through inlet 23 by the Venturi-action of the gas stream passing through the channels 25. Part of this carrier stream can be ionized by the discharge to permit focusing or defocusing of the beam. When the rod material erodes at a relatively high rate, the current through the secondary winding 31 is relatively high so that the voltage drop supplied to the servomotor 36 via current transformer 34 is likewise high, the resulting unbalance of the servomotor 36 causing an angular displacement of its rotor to open valve 28 and increase the carrier-gas flow through the nozzles. When the erosion rate is relatively small, a reverse sequence of events occurs.

In FIG. 2 I show a modification of the device of FIG. 1 wherein the electrodes 115, 116 are again energized by means of a transformer 131 from a high-frequency generator 133 but wherein a focusing coil 140 is provided in the region of the spark gap to concentrate the beam. A carrier-gas flow of the type described with reference to FIG. 1 can also be carried out in this system. The electromagnetic control means illustrated in FIG. 2 includes an alternating-current source 141, which may be connected in step with source 133 or be of an entirely different frequency, is bridged across a portion of coil 140 whose entire length is connected across a capacitor 142 forming with the coil a parametric oscillator capable of applying focusing pulses to the beam in step with the eroding discharge. It should be noted that similar results can be obtained if the alternating-current source 141 is dispensed with since the beam 143 of metal particles is at least partly ionized and acts as a moving electrical conductor capable of inducing an electromotive force in coil 140 to charge the capacitor 142 which can constitute a resonant network therewith. It will be apparent that this effect results in the induction of electromagnetic focusing pulses without need for an auxiliary A-C source.

In the embodiment of FIG. 3, the high-frequency current for providing the spark-discharge derives from a series-resonant network 230a, 230b connected across the electrode rods 215, 216. A further series-resonant network 245 is also connected across these rods as a pulse-modifying system. The inductance 230a of the first-mentioned resonant network is air-coupled with the focusing coil 240 by means of a secondary winding 230c, the focusing coil 240 being parametrically energized by a capacitor 242 as previously described. A two-position switch 246 is provided to permit selective energization of the electrodes by a battery 247, representative of any direct-current source, via a pulse-shaping choke 248 or the high-frequency A-C source 233 via capacitor 249.

Example

In an apparatus of any of the general types shown in FIGS. 1-3, copper can be sprayed upon a synthetic resin substrate. In this case, both electrodes 15, 16; 115, 116; and 215, 216 are composed of copper while the substrate is a melamine resin positioned 30 cm. from the nozzle. The carrier gas is ionized air which is supplied to the

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nozzle at a pressure of 5 kg. per cm.² with a flow velocity of 20 meters per second. The metal particle beam strikes the plastic at a temperature of about 150° C. after generation by a spark discharge of 100 amps with a potential of 50 volts. A discharge frequency of 10 kilocycles per second was used in the deposition voltage and the desired thickness of metal on the substrate was achieved. It was possible, in this manner, to deposit metals to a uniform depth of several microns within 5 seconds. The frequency of the focusing current applied to the focusing coils 140, 240 was also 10 kc./sec.

In FIG. 4 there is shown a modified apparatus whereby a layer 311 can be applied on a body 310. In this case, a funnel-shaped nozzle 324 opens in the direction of this body 310. This nozzle surrounds an inner nozzle 312 with clearance so that channels 325 are formed between the connecting webs. A rod 315 of the material to be deposited passes axially into the nozzles 312, 324 and has drive rollers 350 rotated by a motor 351 for feeding this rod progressively into the inner nozzle 312. The servomotor 351 also drives a damper 328 forming flow-control means in the supply conduit 327 by means of which a carrier gas is fed through channels 325 of the nozzles. In this case a main induction coil 352 serves to heat the rod 315 and is energized by a high-frequency alternating current source 353, the induction coil 352 being connected with an auxiliary induction coil 354 in parametric relationship with the main induction coil and provided with capacitor 342. The auxiliary coil 354 can be so poled to act as a focusing magnet in the manner previously described or merely as an added high-frequency heating element. In any event, the rod 315 is preferably composed of a magnetically permeable or ferrous metal. A high-voltage energizing coil 355 is disposed coaxially with coils 352 and 354 around the rod 315 rearwardly of the main coil and activates a pair of arc discharge electrodes 355', 355'' to produce a corona discharge capable of converting oxygen in the gas passing around rod 315 into charged particles of ozone, the electric current passing through this coil being derived solely by induction from the moving magnetically permeable rod 315.

As can be seen in FIG. 4 the energizing circuit for the main induction coil 352 includes a shunt 334, which functions in the manner of a current transformer to produce a voltage drop across the high-ohmic potentiometer 334a which is tapped to feed a servo-amplifier 358. This amplifier includes a transistor 359 whose emitter-base network includes the tapped sensing means 334, 334a and a biasing resistor 360. The emitter-collector circuit includes a direct-current source 361 in series with a resistor 362 and an ammeter 363 providing a visual indication of the current flow through the indication-heating circuit. The armature 351a of servomotor 351 is connected in series with a battery 364 across the load-resistor 362. Resistor 362, in effect, serves as a comparing element in which the current passing through the armature 358 normally bucks the current from transistor 359. When the induction current drops to reduce the rate of erosion of the rod 315, the control device 358 causes a rotation of armature 351a in a sense tending to decrease the gas flow through the nozzle. The flared configuration of the latter results in a highly divergent spray cone 343 suitable for rapid coverage of wide areas with thin layers.

In the modification shown in FIG. 5 corona screens 355', 355'' are energized by being bridged across the series combination of the main heating portion 352a and the corona-activating portion 355a of the induction coil. The auxiliary coil 354a is here connected to a tap of the main induction coil 352a in an oppositely effective direction. The energizing circuit for the coils 352a, 354a and 355a includes the A-C source 353a and capacitor 342a, the amplifier 358 being provided as in the device of FIG. 4 to control the carrier fluid.

It should be noted that the "focusing" action discussed above includes a radial component of magnetic force tending to compress the ionized stream as well as an axial component which increases the kinetic energy of the particles impinging upon the substrate.

The invention described and illustrated is believed to admit of many modifications and variations within the ability of persons skilled in the art, all of these modifications and variations being considered within the spirit and scope of the appended claims.

What is claimed is:

1. A method of spray depositing particles of a first substance upon a substrate, comprising the steps of forming said particles of said first substance by heating a body thereof; entraining said particles of said first substance in a stream of gaseous particles of a second substance by passing said gas stream around the heated body; directing said gas stream against said substrate to form a coating of said first substance thereupon; ionizing at least some of the particles of at least one of said substances by effecting an electrical space discharge in said stream to impart electrical charge thereto; subjecting said gas stream to a magnetic field, thereby controlling the configuration of said stream; electromagnetically deriving a control signal from a parameter of the electrically charged stream downstream of said space discharge and regulating at least one of the following variables with said control signal: (a) the rate of flow of said gas stream, (b) the rate of ionization of said particles, (c) the rate of heating of said body, and (d) the intensity of said magnetic field.

2. A method of spray depositing particles of a first relatively electropositive metallic substance upon a substrate, comprising the steps of forming said particles of said first substance by heating a body thereof; entraining said particles of said first substance in a stream of gaseous particles of a second, relatively electronegative substance by passing said gas stream around the heated body; directing said gas stream against said substrate to form a coating of said first substance thereupon; ionizing at least some of the particles of at least one of said substances by effecting an electrical space discharge in said stream to impart electrical charge thereto; subjecting said gas stream to a magnetic field applying electromagnetic force to at least the ionized particles of said gas stream by passing said stream axially through an electrically energized coil, thereby controlling the configuration of said stream; electromagnetically deriving a control signal from a parameter of the electrically charged stream downstream of said space discharge and regulating at least one of the following variables with said control signal: (a) the rate of flow of said gas stream, (b) the rate of ionization of said particles, (c) the rate of heating of said body, and (d) the intensity of said magnetic field.

3. A method of spray depositing particles of a first substance upon a substrate, comprising the steps of forming said particles of said first substance by heating a body thereof; entraining said particles of said first substance in a stream of gaseous particles of a second substance by passing said gas stream around the heated body; directing said gas stream against said substrate to form a coating of said first substance thereupon; ionizing at least some of the particles of at least one of said substances to impart electrical charge to said stream; subjecting said gas stream to a magnetic field, thereby controlling the configuration of said stream; electromagnetically detecting a parameter of said gas stream downstream of the ionization of said particles; and regulating the velocity of said gas stream in step with the rate of formation of said particles of said first substance upon the heating of said body and in dependence upon the electromagnetic detection of said parameter.

4. A method of spray depositing particles of a metallic substance upon a substrate, comprising the steps of forming said particles of said first substance by effecting an

iterative electric spark discharge at a body of said first substance to erode said particles therefrom; entraining said particles of said first substance in a stream of gaseous particles of a second substance by passing said gas stream around said body at least in the region of said discharge; directing said gas stream against a substrate to form a coating of said first substance thereon; ionizing at least some of the particles of at least one of said substances by effecting an electric space discharge in said stream to impart electrical charge thereto; and subjecting said gas stream to a magnetic field by passing said stream axially through a coil electrically energized in step with said spark discharge, thereby controlling the configuration of said stream; electromagnetically deriving a control signal from a parameter of the electrically charged stream downstream of said space discharge and regulating at least one of the following variables with said control signal: (a) the rate of flow of said gas stream, (b) the rate of ionization of said particles, (c) the rate of heating of said body, and (d) the intensity of said magnetic field.

5. A method of spray depositing particles of a metallic substance upon a substrate, comprising the steps of forming said particles of said first substance by heating a body of said first substance to erode said particles therefrom; entraining said particles of said first substance in a stream of gaseous particles of a second substance by passing said gas stream around said body at least in the region of directing said gas stream against a substrate to form a coating of said first substance thereon; ionizing at least some of the particles of at least one of said substances by effecting an electric space discharge in said stream upstream of said region to impart electrical charge to said stream; subjecting said gas stream to a magnetic field by passing said stream axially through an electrically energized coil, thereby controlling the configuration of said stream; electromagnetically deriving a control signal from a parameter of the electrically charged stream downstream of said space discharge and regulating at least one of the following variables with said control signal: (a) the rate of flow of said gas stream, (b) the rate of ionization of said particles, (c) the rate of heating of said body, and (d) the intensity of said magnetic field.

6. Apparatus for the spray deposition of particles of a first substance upon a substrate, comprising conduit means opening in the direction of said substrate; spark discharge means for producing particles of said first substance into said conduit; means for introducing gaseous particles of a carrier fluid into said conduit for entraining said particles of said first substance in a gas stream toward said substrate, thereby forming a coating of said first substance thereon; means in said conduit including said spark-discharge means for ionizing at least some of said particles to impart electrical charge to said stream; means for regulating the flow of said carrier fluid in step with the rate of production of said particles of said first substance; a magnetic coil surrounding the stream downstream of said spark-discharge means for regulating the configuration of the gas stream directed against said substrate; means for energizing said magnetic coil in step with the rate of production of said particles of said first substance; and means downstream of the spark-discharge means for electromagnetically responding to a parameter of the ionized stream and controlling the formation thereof.

7. Apparatus for the spray deposition of particles of a first substance upon a substrate, comprising conduit means opening in the direction of said substrate; means for introducing particles of said first substance into said conduit; means for introducing gaseous particles of a carrier fluid into said conduit for entraining said particles of said first substance in a gas stream toward said substrate, thereby forming a coating of said first substance thereon; electric discharge means in said conduit for ionizing at least some of said particles to impart electrical charge to said stream; control means including a magnetic

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coil surrounding the stream downstream of said electric-discharge means for regulating the configuration of the gas stream directed against said substrate; and means for electrically energizing said magnetic coil including a source of electric current bridged across part of said coil and a capacitor bridged across a further part thereof.

8. Apparatus for the spray deposition of particles of a first substance upon a substrate, comprising conduit means opening in the direction of said substrate; means for introducing particles of said first substance into said conduit; means for introducing gaseous particles of a carrier fluid into said conduit for entraining said particles of said first substance in a gas stream toward said substrate, thereby forming a coating of said first substance thereon; electric discharge means in said conduit for ionizing at least some of said particles to impart electrical charge to said stream; control means including a magnetic

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coil surrounding the stream downstream of said electric discharge means for regulating the configuration of the gas stream directed against the substrate; and an energizing coil inductively responding to the field of the first-mentioned coil and coupled with said electric-discharge means for operating same.

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