United States Patent

[11] **3,613,633**

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[54] METHOD AND APPARATUS FOR COATING ARTICLES UTILIZING ROTATING CRUCIBLE COATING APPARATUS INCLUDING A CENTRIFUGAL-TYPE CRUCIBLE

10 Claims, 9 Drawing Figs.

[52] **U.S. Cl.** .... **118/5,** 118/49.5, 118/59, 118/503, 214/16 B, 219/273, 279/1
[51] **Int. Cl.** **B05c 11/14**
[50] **Field of Search** 118/4, 5, 2, 47–49.5, 500, 503; 219/273, 275

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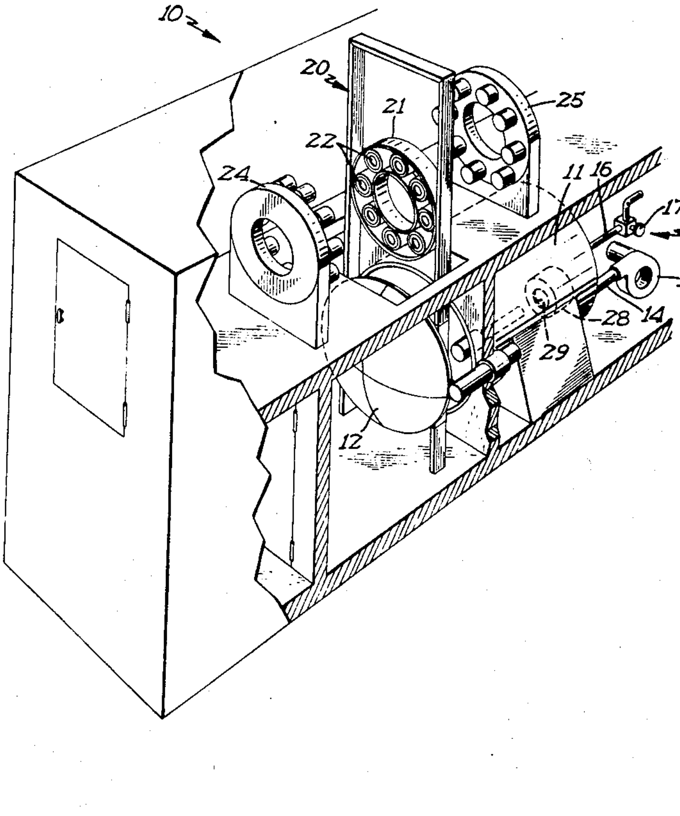
Attorney—Orrin M. Haugen

ABSTRACT: An evaporative deposition system including a chamber having means for evacuation and control of atmosphere, an evaporant-retaining crucible within said chamber having a generally closed cylindrical surface with a generally centrally disposed axis, the crucible having in-turned edge surfaces at either end thereof. Means are provided for heating the crucible, with the heating means being disposed generally radially outwardly of the closed cylindrical surface. In addition, means are provided for rotating the crucible about the central axis at an arcuate rate for centrifugally maintaining evaporant adjacent the inner surface of said closed cylindrical surface, and means are provided for inserting and retaining an article to be coated within said closed cylindrical surface. The article inserting and retaining means holds the article being coated in "sight" of the evaporant, and is provided with thermal control means for controlling substrate temperature, the control means maintaining contact with an interior surface of the article being coated.



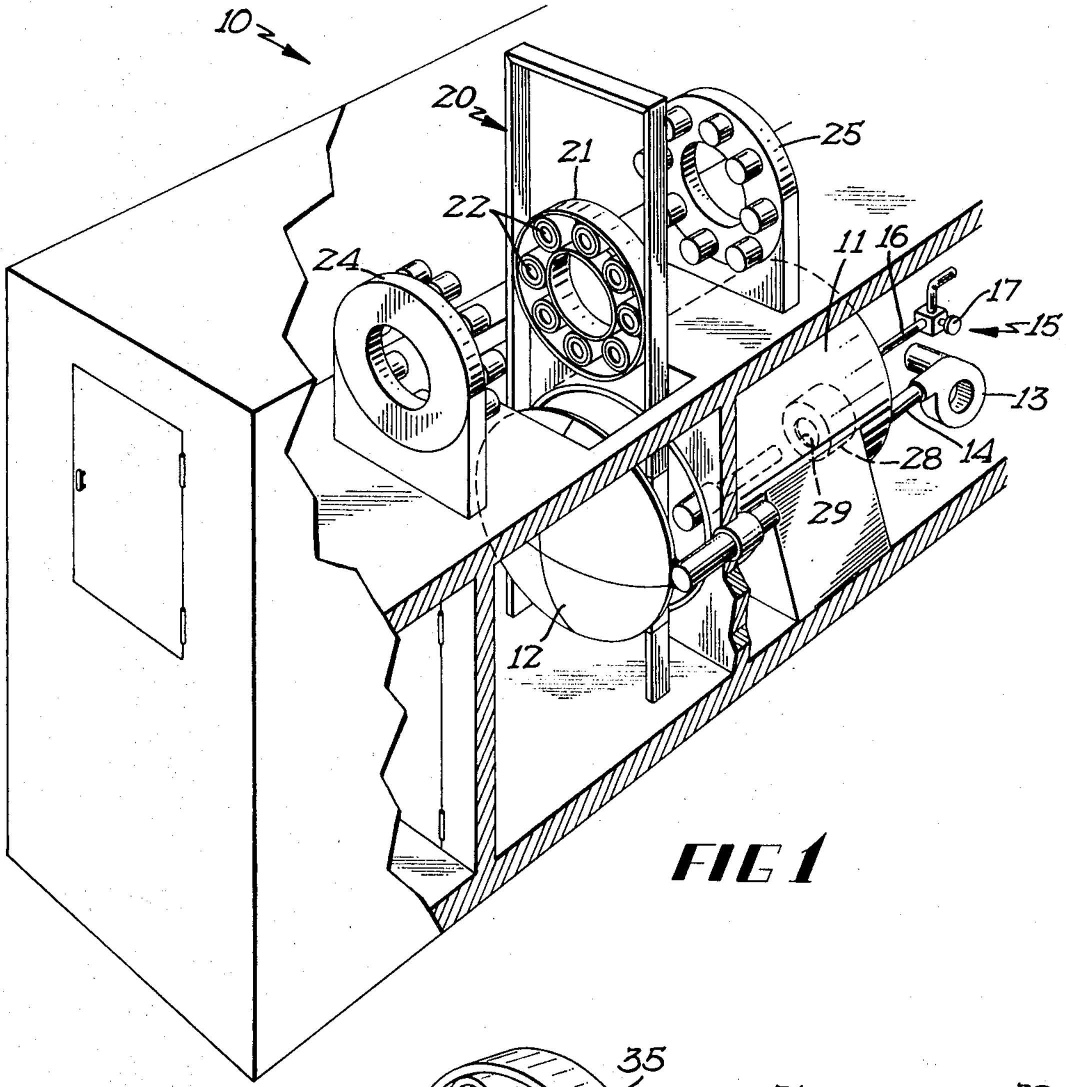


FIG 1

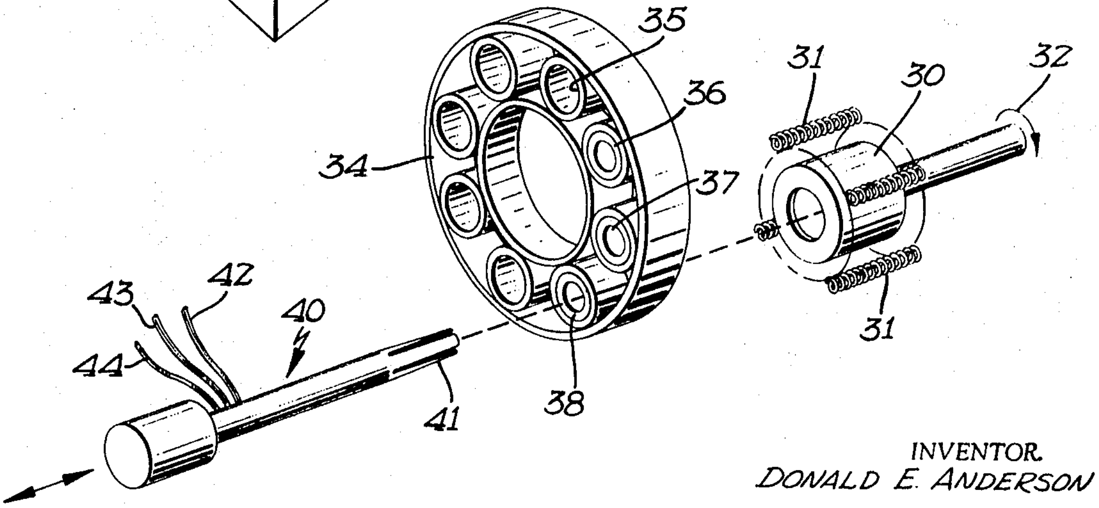


FIG 2

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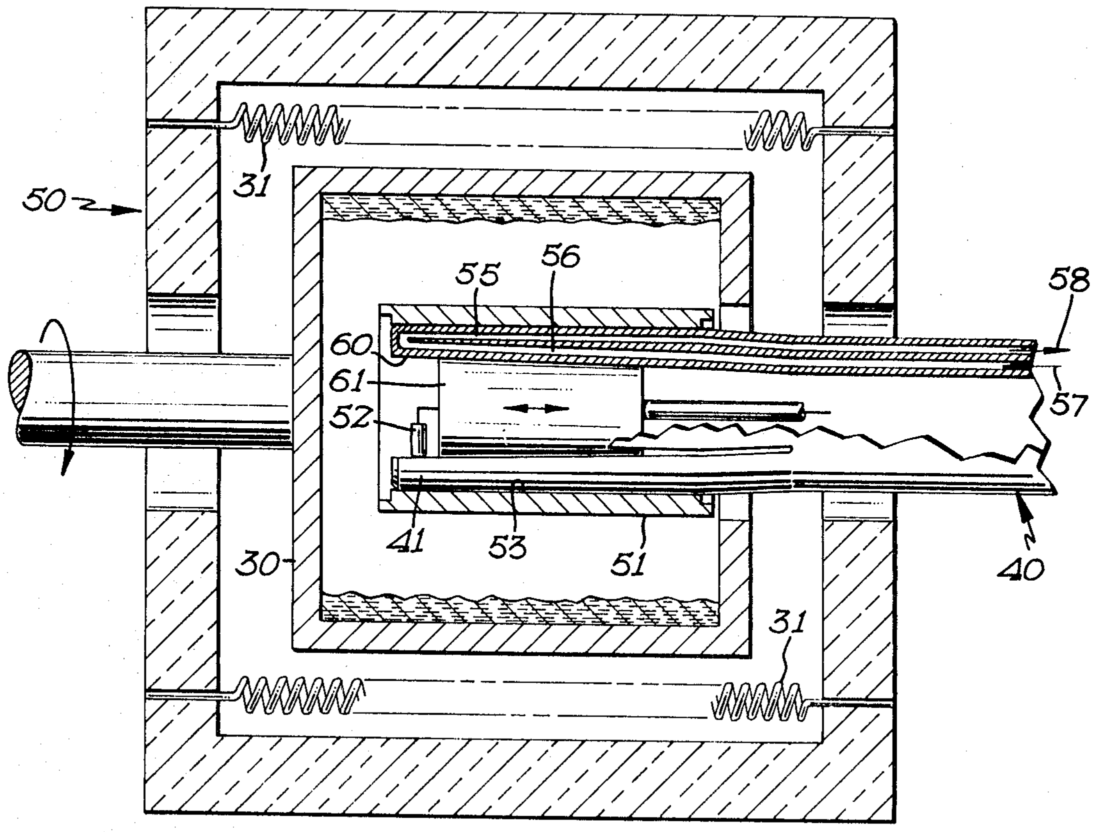

FIG 3

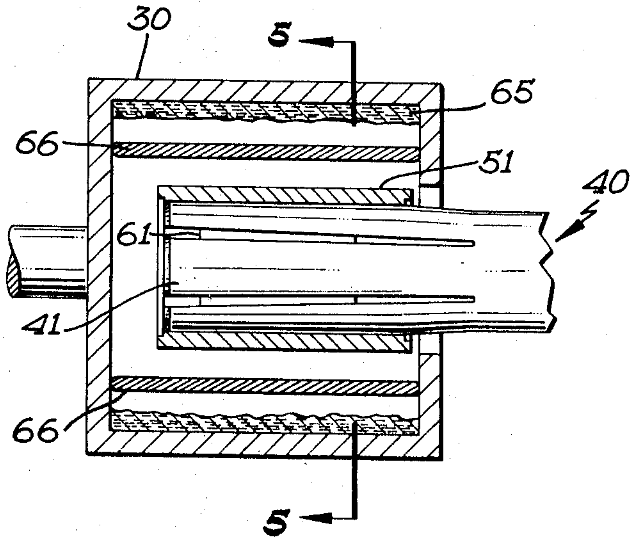

FIG 4

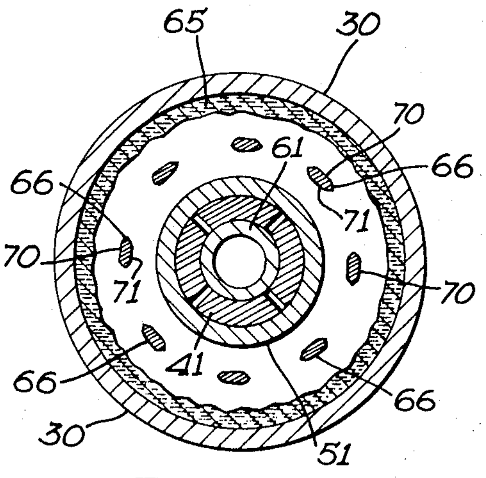

FIG 5

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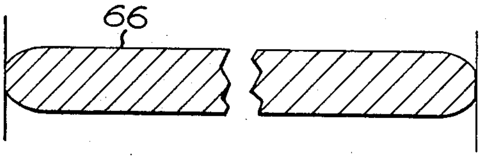

FIG 6

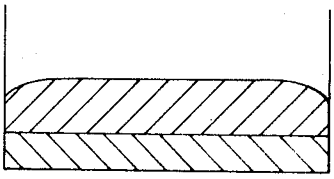

FIG 7a

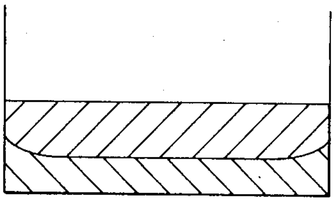

FIG 7b

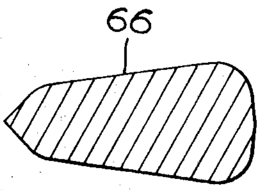

FIG 8

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METHOD AND APPARATUS FOR COATING ARTICLES UTILIZING ROTATING CRUCIBLE COATING APPARATUS INCLUDING A CENTRIFUGAL-TYPE CRUCIBLE

The present invention relates generally to an improved evaporative deposition system, and more specifically to an improved evaporative deposition system which provides for the rapid, uniform, and isothermal deposition of a film on the surface of a substrate member. The apparatus of the present invention is capable of the application of films at high production rates, and in a reproducible fashion.

In the past, evaporative deposition techniques have been utilized for a variety of articles, the systems normally including a stationary crucible and a stationary substrate. Where unusual configurations are considered, means are provided for moving the substrate in the hope of achieving uniformity in the deposited film.

In accordance with the present invention, however, a crucible is provided which is in the form of a right circular cylinder, the crucible being heated from an externally disposed source, and being arranged to be rotated about its central axis. The rate of rotation is sufficient to provide for at least 1 g. of centrifugal force so as to eliminate the danger of splattering of evaporant. The substrate being coated is mounted on a suitable collet or other device for insertion into the rotating crucible, and for retention therein during the coating operation. Normally, it is desirable to provide thermal control means which cooperate with the collet for maintaining contact with the article being coated, the contact being along a surface which is concealed from the evaporant. Upon completion of the coating operation, the article is removed from the chamber. If desired, baffle or shutter means may be utilized to prevent evaporative deposition from occurring during the insertion and extraction operations, or, as an alternative, suitable tapered baffle means may be utilized to accommodate a specifically programmed insertion and extraction operation.

In the preparation of films of uniform thickness along the Z-axis, large source-to-substrate spacings are normally required. The present arrangement provides a system for eliminating this requirement of large spacing, and high production rates and reproducible devices may accordingly be fabricated. In this connection, deposition occurs uniformly on the entire surface of the substrate, and not merely upon certain preselected surface areas, thereby affording better control of the entire system. Also, with a rigid nonrotating support collet, temperature monitoring and control of the substrate is greatly simplified.

The rate of deposition is primarily a function of source temperature, and hence localized "hot spots" within the crucible may produce inhomogeneous or anomalous deposits to occur. The rotating crucible provides a high degree of temperature "leveling" of the source, and thus the resultant film is highly uniform an homogeneous. By controlling the distance between the surface of the source and the surface of the substrate to a distance which is less than the mean free path of the evaporant within the system, relatively large changes in evaporant depth or reserve may be provided for in the crucible so as to accommodate sequential deposition onto many substrate members.

Therefore, it is a primary object of the present invention to provide for an improved system for the preparation of evaporatively deposited films, wherein the films may be rapidly, uniformly, and isothermally deposited at high production rates, and in a reproducible fashion.

It is yet a further object of the present invention to provide an improved system for the preparation of evaporatively deposited films, the system employing a rotating crucible which moves relative to a heating source, so as to provide temperature leveling in the crucible, and accordingly temperature leveling of the source.

It is yet a further object of the present invention to provide an improved system for the preparation of evaporatively deposited films, wherein a rotating crucible is employed in combination with a substrate retaining member which is nonrotating, and which is closely thermally controlled.

Other and further objects of the present invention will become apparent to those skilled in the art upon a study of the following specification, appended claims, and accompanying drawings wherein:

FIG. 1 is a perspective view of a typical installation system employing the deposition apparatus of the present invention;

FIG. 2 is a detail perspective view of the substrate-retaining collet, a substrate-retaining magazine, and the rotating crucible apparatus, all of which are arranged in accordance with the present invention;

FIG. 3 is a sectional view of the coating system prepared in accordance with the present invention, and illustrating the crucible oven, rotating crucible, and support collet useful in the systems shown in **FIGS. 1** and **2**; **FIG. 4** is a detail sectional view similar to **FIG. 3**, an illustrating the structure with shutter blades or baffles utilized in combination with the rotating crucible and source;

FIG. 5 is a vertical sectional view taken along the line and in the direction of the arrows **5—5** of **FIG. 4**;

FIG. 6 is a detailed sectional view showing a preferred configuration or profile for the individual shutter blades; and

FIGS. 7A and **7B** illustrate the uncorrected and corrected deposition profile which is achieved through the utilization of a blade configuration such as is shown in **FIG. 6**; and

FIG. 8 is a vertical sectional view taken along the longitudinal axis of the shutter blades shown in **FIGS. 4, 5** and **6**.

In accordance with the preferred modification of the present invention, and with particular attention being directed to **FIG. 1**, of the drawings, it will be seen that the vacuum deposition system generally designated **10** includes a vacuum chamber **11** in the form of a right circular cylinder, along with an opening door member **12** for controlling access to the system. A vacuum pump is shown at **13**, the pump being coupled to the chamber by the conduit **14**. Also, a vent system for controlling the atmosphere within the chamber is shown generally at **15**, and includes a suitable conduit communicating with the chamber, as at **16**, along with a suitable control valve **17**.

Adjacent the opening to the chamber, an elevator arrangement is shown as at **20**, the elevator being arranged to handle a rotary magazine element or the like **21**, and raise and lower the individual articles to be coated, such as the right circular cylindrical members **22—22**. A loading fixture is illustrated at **24**, this fixture being utilized to transfer the individual articles being coated, such as the articles **22—22**, from the magazine member **21** to the unloading fixture **25**. The system may accordingly be rendered automatic, or semiautomatic, depending upon the requirements of the apparatus and the material being deposited. For example, certain toxic materials such as, for example, selenium, tellurium, or others, may be handled in these systems only with proper safeguards for the operating personnel, and in that case, a closable access hatch will be provided in the area of the elevator system **20**, to separate effectively or isolate the upper materials-handling zone from the zone housing the vacuum chamber.

With attention being redirected to the chamber **11**, it will be seen that a coating crucible is confined within the chamber, such as at **28**, this chamber being provided with a substrate access opening such as at **29**, this opening being utilized to permit the insertion and extraction of individual articles from the interior of the member **28**. As is illustrated in **FIGS. 3** and **4**, the member **28** is provided with suitable heating elements for the crucible.

Attention is now directed to FIG. 2 of the drawings wherein certain features of the system employing the aspects of the present invention are shown. In this view, a rotating crucible member is shown at **30**, with suitable heaters **31** disposed outwardly of the outer surface of the crucible **30**. The crucible **30** is arranged to rotate in accordance with the arrow shown at **32**. If desired, heat shields may be provided outwardly form the heaters, in order to more carefully control the temperature of the crucible **30** and its neighboring crucibles, if present.

The magazine **34** is provided with a plurality of chambers or the like as at **35**, these chambers being disposed radially outwardly of the center of the magazine, and accordingly capable of receiving individual articles to be coated. In the illustration of FIG. **2**, the articles being coated are in the form of right circular cylinders which may, for purposes of illustration and explanation, be hollow sleeve elements. The individual articles to be coated are shown in the chamber as at **36**, **37**, and **38**.

Attention is now directed to the means for inserting and retaining the article to be coated, such as the axially movable collet member generally designated **40**. This collet member has an expandable tip portion as at **41**, this tip portion **41** having individual temperature sensors disposed adjacent the external surface thereof, with sensor lines in communication with the individual sensors, and shown at **42**. Coolant is passed through the individual surfaces of the collet, with coolant fluid being supplied through conduit **43**, and passed outwardly through conduit **44**. In this fashion, thermal control of the substrate may be accomplished, thereby contributing to uniformity and reproducibility in the finished product.

In the operation of that portion of the system shown in FIG. **2**, the expandable collet **41** is inserted into the confines of one of the hollow drums retained within the magazine, such as the drum **38**, and upon continued forward axial motion of the collet **41**, the hollow drum **38** is transferred to the confines or interior of rotating crucible **30** where the deposition of the film or coating occurs.

Attention is now directed to FIG. **3** of the drawings wherein the crucible **30** is shown as being retained within a suitable crucible oven. The crucible **30** is fabricated from a suitable refractory material, or metallic structure with a wall thickness capable of withstanding the centrifugal forces encountered, the oven being fabricated from a suitable refractory material or the like. Heaters such as the heating elements **31** are disposed radially outwardly from the outer surface of the rotating crucible **30**, these heaters being either radiantly or inductively coupled to the crucible **30** and its contents, and preferably being arranged in equal arcuately spaced relationship about the crucible. The support collet is shown retaining the hollow drum or sleeve being coated, the article being coated being shown in greater detail in FIGS. **4** and **5**, and will be discussed hereinafter. For example, one form of temperature sensor is shown at **52**, this being a thermocouple which has its hot junction disposed immediately adjacent the surface of the expandable collet. As is apparent from FIG. **5**, the expandable collet utilizes a plurality of individual arcuate segments which can be expanded outwardly to make physical contact with the inner surface **53** of drum **51**, this contact being a good thermally conductive contact in addition to a mechanical contact. Coolant is provided in and along the bores **55** and **56**, with coolant being transmitted through these lines at a relatively high rate. With coolant moving rapidly through these lines, it is possible to monitor the temperature at the surface of the article **51**, without having to experience local warm and local cold areas. The liquid coolant moves in the direction of the arrows shown at **57** and **58**.

In order to provide for expansion and contraction of the collet, the interior of the individual segments forming the collet, such as is shown at **60** is in the form of a cone converging toward the right. The expander utilized in combination with this cone is in the form of a conical ramp shaft **61** which is capable of motion in either of two axial directions, so that upon moving to the right, the collet effectively expands and clamps the individual collet segments against the inner surface of the article to be coated. Motion in the opposite axial direction provides for contraction of the individual collet segments. It will be appreciated, of course, that this expansion and contraction arrangement is typical of only one of a variety of such arrangements which could be used in connection with the operation of the present invention.

Attention is now directed to FIG. **4** of the drawings wherein the rotating crucible **30** is illustrated, the crucible **30** being provided with an evaporant source **65**, along with the article to be coated **51**. Shutter blades are provided as at **66**, these blades being formed with a lenticular profile such as is illustrated in FIG. **5**, for a purpose hereinafter made manifest. These individual shutter blades or baffles have a source-viewing surface **70** which is normally hot, and a relatively cooler substrate-viewing surface **71**. The blades may, of course, be formed with a variety of cross-sectional configurations depending upon the nature of the task being undertaken, however the profile or configuration shown in FIG. **5** is discussed in greater detail in connection with FIGS. 7A and 7B hereinafter.

It is also possible to heat and/or cool individual shutter blades such as the blades **66**, and by the use of such heating or cooling (for example, thermoelectric Joule heat or Peltier cooling) the radial temperature profile can be established for the evaporant and for the substrate. Also, the hot side of the individual blades or baffles can be heated so as The lenticular remain clean, while the cold side will, by virtue of its disposition in the arrangement, remain clean since it receives only a small amount of evaporant which is transmitted or returned from the surface of the cooled substrate.

The lenticular configuration for the cross section of the individual shutter blades is provided in order to permit an effective "throttling" of the deposition rate, thereby permitting a higher temperature in the evaporant, for a given deposition rate. The availability of these shutter elements, therefore, adds certain degrees of freedom for the operator to employ during his coating operation. The lenticular configuration provides for a leveling of the flow of evaporant from the source onto the substrate surface.

Attention is now directed to FIG. **6** of the drawings wherein the axial profile of the individual blades **66** is shown. The reason for the configuration is shown in FIGS. 7A and 7B, wherein the effective source area provides a deposition rate or thickness in the traditional fashion. The end walls of the crucible act as baffles or barrier surfaces, and with the use of blades of the profile shown in FIG. **6**, the effective source area is controlled, along with the deposition rate so as to provide a uniform film thickness along the axial extent of the article being coated.

Better results are also obtained since radiant heat is retained by the crucible in a more uniform fashion. Also, the rate of heat transfer to the substrate is decreased by a factor proportional to the arcuate width of the individual baffles or blades. For a given rate of deposition on a substrate, the total heat transfer to the substrate is actually decreased, this being due to the fact that the rate of evaporation is an exponential function of the temperature, while the radiant gain is the fourth-power dependence of radiation upon temperature. Also, as is illustrated in FIG. **8**, the individual shutter blades or baffles can be shaped in the axial direction to correct the deposition rate for variations near the end walls, or to accommodate the insertion and withdrawal operations and the consequent time variable incurred for the substrate surface.

It is also recognized that with baffles or blades being employed, the evaporation rate can be controlled with a higher source temperature thereby enhancing the uniformity of the product without risking the dangers of uncontrollable substrate temperatures.

The use of the apparatus of the present invention in connection with circular substrates or cylindrical substrates is highly advantageous, since every point on the surface of the substrate views an enclosed confining "wall" of evaporant at a predetermined temperature. Also, the pressure range of the system may be controlled so that the distance between the surface of the substrate and the surface of the evaporant is less than the mean free path of the evaporant in the system. This will assure retention of Knudsen flow, which eliminates molecular interactions in the flow space.

It is normally preferred that the evaporant cover the entire surface of the rotating crucible, thus providing for continuous uniform coating of the substrate with the evaporant. For certain operations, it is also desirable that the flow of coolant be programmed so as to accommodate increases in substrate temperature encountered by latent heat of condensation.

These flow conditions are readily apparent to those skilled in the art, and a given system will be susceptible of uniform and precise control.

In the evaporative deposition of certain materials, it is not necessary to achieve the liquid estate for the evaporant source. Certain materials can have a sufficiently high vapor pressure under operating conditions so that sublimation will occur, thereby achieving an evaporative deposition without requiring the maintaining of the liquid evaporant on the walls by centrifugal force. It is also important to note that the large supply of evaporant will permit a significant number of substrates to be coated without recharging the crucible. The area relationships are, of course, apparent from a review of the structure shown in FIG. 3. Also, with continued attention being directed to FIG. 3, it will be observed that the opening formed in the rotating crucible **30** is substantially the same as the outer diameter of the cylindrical substrate **51**, the clearance being modest for the purpose of retaining evaporant within the confines of the chamber of rotating crucible **30**. This feature will, of course, prevent evaporant from leaving the chamber in an uncontrolled or uncontrollable fashion. In certain instances, it may be desirable to insert a baffle or gland in the system in order to prevent loss of evaporant during the deposition operation. Such baffles are desired when toxic materials are being treated, such as, for example, selenium or tellurium.

What is claimed is:

1. In an evaporative deposition system:

a. a vacuum chamber having means for evacuation and for control of residual atmosphere and pressure;

b. a crucible within said chamber for retaining evaporant and having a generally closed cylindrical surface with a generally centrally disposed axis, and in-turned end surfaces at either end of said cylindrical surface;

c. means for heating said crucible disposed generally radially outwardly of said closed cylindrical surface and arranged for heating said crucible and evaporant;

d. means for rotating said crucible about the centrally disposed axis thereof at an arcuate rate for centrifugally maintaining evaporant adjacent the inner surface of said closed cylindrical surface;

e. means for inserting and retaining an article to be coated within said closed cylindrical crucible, at least one surface of said article being in sight of said retained evaporant;

f. thermal control means for cooperating with said insertion and retention means for maintaining contact with a second surface of said article to be coated, said second surface being concealed from said retained evaporant.

2. The evaporative deposition system as defined in claim **1** being particularly characterized in that said thermal control means is adapted to cool the article to be coated.

3. The evaporative deposition system as defined in claim **1** being particularly characterized in that said means for inserting and retaining the article to be coated is an expandable support collet.

4. The evaporative deposition system as defined in claim **1** being particularly characterized in that a plurality of baffles are mounted within said crucible and disposed radially outwardly of the axis thereof for controlling the radially inwardly directed flow of evaporant from the outer surfaces of said closed cylindrical surface.

5. The evaporative deposition system as defined in claim **4** being particularly characterized in that said baffles have a lenticular cross Section.

5. The evaporative deposition system as defined in claim **4** being particularly characterized in that said baffles are equally arcuately spaced, one from another, and equally radially spaced relative to said centrally disposed axis.

7. The evaporative deposition system as defined in claim **4** being particularly characterized in that said baffles are movable to effectively close the path between the inner surface of said cylindrical crucible and the outer surface of said collet.

8. The evaporative deposition system as defined in claim **1** being particularly characterized in that said means for insertion and retention of said article being coated includes means for with drawing said article from said chamber, said means for insertion and retention being in the form of a cylindrical collet having an axis arranged generally coaxially with the axis of said crucible.

9. The evaporative deposition system as defined in claim **8** being particularly characterized in that the outer surface of said collet is adapted to be cooled and maintained at a controllable temperature.

10. The evaporative deposition system as defined in claim **1** being particularly characterized in that rotary magazine means are provided for retaining individual articles to be coated prior to insertion into said crucible.

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,613,633 Dated October 19, 1971

Inventor(s) Donald E. Anderson

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 3, line 40, after the word "shown" insert -- here at 51. The features of the expandable collet are shown --.

Column 4, line 16, after the word "as", insert -- to -- and delete the words "The lenticular".

Signed and sealed this 21st day of March 1972

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents