

(19) World Intellectual Property
Organization
International Bureau



(43) International Publication Date
17 November 2005 (17.11.2005)

PCT

(10) International Publication Number
WO 2005/109963 A1

(51) International Patent Classification⁷: **H05B 33/10**,
C23C 14/24

(21) International Application Number:
PCT/US2005/014888

(22) International Filing Date: 29 April 2005 (29.04.2005)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
10/837,190 30 April 2004 (30.04.2004) US

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(81) Designated States (unless otherwise indicated, for every
kind of national protection available): AE, AG, AL, AM,
AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN,
CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI,
GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE,
KG, KM, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA,
MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM,
PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY,
TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU,
ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every
kind of regional protection available): ARIPO (BW, GH,
GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM,
ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM),
European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI,
FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO,
SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN,
GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— with international search report

For two-letter codes and other abbreviations, refer to the "Guid-
ance Notes on Codes and Abbreviations" appearing at the begin-
ning of each regular issue of the PCT Gazette.

(54) Title: DEPOSITION APPARATUS FOR TEMPERATURE SENSITIVE MATERIALS

(57) Abstract: A system for the deposition of vaporized materials on a substrate is described, comprising at least first and second orientation-independent apparatuses for directing vaporized organic materials onto a substrate surface to form first and second films, each of the first and second orientation-independent apparatuses being arranged in a different relative orientation and comprising: a chamber containing a quantity of material; a permeable member at one end of the chamber with a heating element for vaporizing the material; and a piston at the other end of the chamber for continuously feeding the material toward the permeable member as it is vaporized, whereby organic material vaporizes at a desired rate-dependent vaporization temperature at the one end of the chamber. A plurality of thin films may be deposited on a substrate using deposition apparatus in a variety of orientations. Such a design provides reduced costs and improved deposition rate control.



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DEPOSITION APPARATUS FOR
TEMPERATURE SENSITIVE MATERIALS

FIELD OF THE INVENTION

5 The present invention relates to the field of physical vapor deposition where a source material is heated to a temperature so as to cause vaporization and produce a vapor plume to form a thin film on a surface of a substrate.

BACKGROUND OF THE INVENTION

10 An OLED device includes a substrate, an anode, a hole-transporting layer made of an organic compound, an organic luminescent layer with suitable dopants, an organic electron-transporting layer, and a cathode. OLED devices are attractive because of their low driving voltage, high luminance, wide-
15 angle viewing and capability for full-color flat emission displays. Tang et al. described this multilayer OLED device in their U.S. Patents 4,769,292 and 4,885,211.

 Physical vapor deposition in a vacuum environment is the principal way of depositing thin organic material films as used in small molecule OLED
20 devices. Such methods are well known, for example, Barr in U.S. 2,447,789 and Tanabe et al. in EP 0 982 411. The organic materials used in the manufacture of OLED devices are often subject to degradation when maintained at or near the desired rate-dependent vaporization temperature for extended periods of time. Exposure of sensitive organic materials to higher temperatures can cause changes
25 in the structure of the molecules and associated changes in material properties.

 To overcome the thermal sensitivity of these materials, only small quantities of organic materials have been loaded in sources, and they are heated as little as possible. In this manner, the material is consumed before it has reached the temperature exposure threshold to cause significant degradation. The
30 limitations with this practice are that the available vaporization rate is very low

due to the limitation on heater temperature, and the operation time of the source is very short due to the small quantity of material present in the source. The low deposition rate and frequent source recharging place substantial limitations on the throughput of OLED manufacturing facilities.

5 A secondary consequence of heating the entire organic material charge to roughly the same temperature is that it is impractical to mix additional organic materials, such as dopants, with a host material unless the vaporization behavior and vapor pressure of the dopant is very close to that of the host material. This is generally not the case and, as a result, prior art devices frequently require
10 the use of separate sources to co-deposit host and dopant materials.

 The organic materials used in OLED devices have a highly non-linear vaporization rate dependence on source temperature. A small change in source temperature leads to a very large change in vaporization rate. Despite this, prior-art devices employ source temperature as the only way to control
15 vaporization rate. To achieve good temperature control, prior-art deposition sources typically utilize heating structures whose solid volume is much larger than the organic charge volume, composed of high thermal-conductivity materials that are well insulated. The high thermal conductivity insures good temperature uniformity through the structure, and the large thermal mass helps to maintain the
20 temperature within a critically small range by reducing temperature fluctuations. These measures have the desired effect on steady-state vaporization rate stability but have a detrimental effect at start-up. It is common that these devices must operate for many hours at start-up before steady-state thermal equilibrium and hence a steady vaporization rate is achieved.

25 A further limitation of prior-art sources is that the geometry of the vapor manifold changes as the organic material charge is consumed. This change requires that the heater temperature change to maintain a constant vaporization rate, and it is observed that the plume shape of the vapor exiting the orifices changes as a function of the organic material thickness and distribution in the

source. Moreover, the structural design of prior-art sources limits the orientation of the vapor plumes. This in turn reduces the variety of deposition systems to which the prior-art sources may be applied.

As noted above, reducing the thermal load of the materials prior to deposition contributes to the longevity of the materials. Since the materials are solids, chambers containing the materials will empty as the materials are vaporized. Typically, material is vaporized from a top surface. If the material is not held within the chamber such that the sublimating top surface is physically above the remainder of the material, the sublimated material will not form a well-controlled plume and material may even fall out of the chamber. Hence, the geometry of the prior-art sources limits the vapor plume orientation.

For example, WO2003062486 A1 entitled "Linear or Planar type Evaporator for the Controllable Film Thickness Profiled" describes an evaporator for evaporating and depositing a source material on a substrate located over the evaporator. In an alternative design for evaporating material onto a vertical surface, DE 101 28 091 C 1 entitled "Vorrichtung für die Beschichtung eines flächigen Substrats" by Hoffmann et al., illustrates a vertical deposition source using an angle tube into which the material is deposited. Yet another alternative for vertical deposition is disclosed in WO2003079420 A1 entitled "Evaporation Source for Deposition Process and Insulation Fixing Plate, and Heating Wire Winding Plate and Method for fixing Heating Wire" by LEE et al. This invention discloses a linear evaporation source used on its side. However, neither of these designs can be used in alternative orientations and are therefore limited in their applicability.

US6367414 B2 entitled "Linear aperture deposition apparatus and coating process" by Witzman, et al describes a linear aperture deposition apparatus and process that includes a source box containing a source material, a heating element to sublime or evaporate the source material, and a chimney to direct the source material vapor from the source box to a substrate. A flow restricting baffle having a plurality of holes is positioned between the source material and the

substrate to confine and direct the vapor flow, and an optional floating baffle is positioned on the surface of the source material to further restrict the vapor flow, thereby substantially eliminating source material spatter. A variety of designs are disclosed some of which may be employed in a variety of orientations. However,
5 no design may be used in more than one orientation and rely on gravity to provide a suitable material surface for sublimation.

There is a need, therefore, for an improved deposition system and apparatus for temperature-sensitive material that overcomes these objections.

10 SUMMARY OF THE INVENTION

In accordance with one embodiment, the invention is directed towards a system for the deposition of vaporized materials on a substrate, comprising at least first and second orientation-independent apparatuses for directing vaporized organic materials onto a substrate surface to form first and
15 second films, each of the first and second orientation-independent apparatuses being arranged in a different relative orientation and comprising: a chamber containing a quantity of material; a permeable member at one end of the chamber with a heating element for vaporizing the material; and a piston at the other end of the chamber for continuously feeding the material toward the permeable member
20 as it is vaporized, whereby organic material vaporizes at a desired rate-dependent vaporization temperature at the one end of the chamber.

In a further embodiment, the invention is directed towards a method of depositing thin-films on a substrate comprising the steps of: a) providing a substrate; b) providing at least first and second orientation-
25 independent material vaporization and deposition apparatuses; c) continuously moving the substrate past the first and second orientation-independent apparatuses; and d) directing vaporized organic materials in distinct relative directions from each of the first and second orientation-independent apparatuses and coating thin films of vaporized material on the substrate.

In yet a further embodiment, the invention is directed towards an orientation-independent apparatus for vaporizing and depositing organic materials onto a substrate surface to form a film, comprising: a chamber containing a quantity of material; a permeable member at one end of the chamber with a single
5 heating element for vaporizing the material at a desired rate-dependent vaporization temperature at the one end of the chamber; and a piston at the other end of the chamber for continuously feeding the material toward the permeable member as it is vaporized.

10

ADVANTAGES

It is an advantage of the present invention that a deposition system for depositing a plurality of thin films on a substrate can use deposition apparatus in a variety of orientations. Such a design provides reduced costs and improved deposition rate control.

15

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a cross-sectional view of a vaporization apparatus which may be employed according to one embodiment of the present invention including a piston and a drive mechanism as a way for metering organic material into a
20 heating region;

FIG. 2 shows a graphical representation of vapor pressure vs. temperature for two organic materials;

FIG. 3 is a cross-sectional view of a vaporization apparatus which may be employed according to another embodiment of the present invention
25 including a hydraulically driven piston;

FIG. 4 is a cross-sectional view of a vaporization apparatus which may be employed according to a third embodiment of the present invention including a single heating region;

FIG. 5 is a schematic illustration of a deposition chamber enclosing
30 a substrate and a vaporization apparatus which may be employed according to an embodiment of the present invention;

FIG. 6 is a cross-sectional view of an OLED device structure that can be prepared with the present invention;

FIG. 7 is a cross-sectional view of a system having a plurality of vaporization apparatuses according to an embodiment of the present invention;

5 FIG. 8 is a cross-sectional view of an alternative system having a plurality of vaporization apparatuses according to an embodiment of the present invention;

FIG. 9 is a cross-sectional view of a vaporization apparatus with a mask and substrate according to an embodiment of the present invention;

10 FIG. 10 is a cross-sectional view of a vaporization apparatus with a mask, substrate, and support according to an embodiment of the present invention;

FIG. 11 is a perspective view of a linear source vaporization apparatus which may be employed according to an embodiment of the present invention;

15 FIG. 12 is a perspective view of a point source vaporization apparatus which may be employed according to an embodiment of the present invention; and

FIG. 13 is a perspective view of a planar source vaporization apparatus which may be employed according to an embodiment of the present invention.
20

DETAILED DESCRIPTION OF THE INVENTION

A system for the deposition of vaporized materials on a substrate includes two or more orientation-independent material vaporization and
25 deposition apparatuses for directing vaporized organic materials onto a substrate surface to form two or more thin-films. Each of the orientation-independent apparatuses are arranged in a different relative orientation and comprise: a chamber containing a quantity of material; a permeable member at one end of the chamber with a heating element for vaporizing the material; and a piston at the

other end of the chamber for continuously feeding the material toward the permeable member as it is vaporized, whereby organic material vaporizes at a desired rate-dependent vaporization temperature at the one end of the chamber.

Turning now to FIG. 1, there is shown a cross-sectional view of one embodiment of an orientation-independent thin-film deposition apparatus of this disclosure. Vaporization apparatus 5 is a device for vaporizing organic materials onto a substrate surface to form a film, and includes a first heating region 25 and a second heating region 35 spaced from first heating region 25. First heating region 25 includes a first heating means represented by base block 20, which can be a heating base block or a cooling base block, or both, and which can include control passage 30. Chamber 15 can receive a quantity of organic material 10. Second heating region 35 includes the region bounded by manifold 60 and permeable member 40, which can be part of manifold 60. Manifold 60 also includes one or more apertures 90. A way of metering organic material includes chamber 15 for receiving the organic material 10, piston 50 for raising organic material 10 in chamber 15, as well as permeable member 40. Vaporization apparatus 5 also includes one or more shields 70.

Organic material 10 is preferably either a compacted or pre-condensed solid. However, organic material in powder form is also acceptable. Organic material 10 can comprise a single component, or can comprise two or more organic components, each one having a different vaporization temperature. Organic material 10 is in close thermal contact with the first heating means that is base block 20. Control passages 30 through this block permit the flow of a temperature control fluid, that is, a fluid adapted to either absorb heat from or deliver heat to the first heating region 25. The fluid can be a gas or a liquid or a mixed phase. Vaporization apparatus 5 includes a way for pumping fluid through control passages 30. Such pumping means, not shown, are well known to those skilled in the art. Through such means, organic material 10 is heated in first heating region 25 until it is a temperature below its vaporization temperature. The vaporization temperature can be determined by various ways. For example, FIG. 2

shows a graphical representation of vapor pressure versus temperature for two organic materials commonly used in OLED devices. The vaporization rate is proportional to the vapor pressure, so for a desired vaporization rate, the data in FIG. 2 can be used to define the required heating temperature corresponding to the
5 desired vaporization rate. First heating region **25** is maintained at a constant heater temperature as organic material **10** is consumed.

Organic material **10** is metered towards permeable member **40** at a controlled rate as the material is vaporized. Preferably, the organic material is also moved at a controlled rate from first heating region **25** to second heating region
10 **35**. Second heating region **35** is heated with a second heating means (not shown), e.g., by a resistive, induction, radiant or RF coupling heating means, and preferably through a resistive wire which heats member **40**, to a temperature above the vaporization temperature of organic material **10**, or each of the organic
15 components thereof. Because a given organic component vaporizes at different rates over a continuum of temperatures, there is a logarithmic dependence of vaporization rate on temperature. In choosing a desired deposition rate, one also determines a necessary vaporization temperature of organic material **10**, which will be referred to as the desired rate-dependent vaporization temperature. The
20 temperature of first heating region **25** is below the vaporization temperature, while the temperature of second heating region **35** is at or above the desired rate-dependent vaporization temperature. In this embodiment, second heating region **35** comprises the region bounded by manifold **60** and permeable member **40**. Organic material **10** is pushed against permeable member **40** by piston **50**, which can be controlled through a force-controlled drive mechanism. Piston **50**, chamber **15**,
25 and the force-controlled drive mechanism comprise a way for metering. This metering means permits organic material **10** to be metered through permeable member **40** into second heating region **35** at a controlled rate that varies linearly with the vaporization rate. Along with the temperature of second heating region **35**, this permits finer rate control of the vaporization rate of organic material **10**
30 and additionally offers an independent measure of the vaporization rate. A thin

cross-section of organic material **10** is heated to the desired rate-dependent temperature, which is the temperature of second heating region **35**, by virtue of contact and thermal conduction, whereby the thin cross-section of organic material **10** vaporizes. In the case where organic material **10** comprises two or more organic components, the temperature of second heating region **35** is chosen to be above the vaporization temperature of each of the components so that each of the organic material **10** components simultaneously vaporizes. A steep thermal gradient on the order of 200°C/mm is produced through the thickness of organic material **10**. This gradient protects all but the immediately vaporizing material from the high temperatures. The vaporized organic vapors rapidly pass through the permeable member **40** and can enter into a volume of heated gas manifold **60** or pass directly on to the target substrate. Their residence time at the desired vaporization temperature is very short and, as a result, thermal degradation is greatly reduced. The residence time of organic material **10** at elevated temperature, that is, at the rate-dependent vaporization temperature, is orders of magnitude less than prior art devices and methods (seconds vs. hours or days in the prior art), which permits heating organic material **10** to higher temperatures than in the prior art. Thus, the two heating zone device and method can achieve substantially higher vaporization rates, without causing appreciable degradation of organic material **10**. The constant vaporization rate, and constant volume of vaporizing organic material **10** maintained in second heating region **35** establish and maintain a constant plume shape. The plume is herein defined as the vapor cloud exiting vaporization device **5**.

Since second heating region **35** is maintained at a higher temperature than first heating region **25**, it is possible that heat from second heating region **35** can raise the temperature of the bulk of organic material **10** above that of first heating region **25**. Therefore, it is necessary that the first heating means can also cool organic material **10** after it rises above a predetermined temperature. This can be accomplished by varying the temperature of the fluid in control passage **30**.

Where a manifold **60** is used, a pressure develops as vaporization continues and streams of vapor exit the manifold **60** through the series of apertures **90**. The conductance along the length of the manifold is designed to be roughly two orders of magnitude larger than the sum of the aperture conductances as described in commonly assigned U.S. Patent Application Serial No. 10/352,558 filed January 28, 2003 by Jeremy M. Grace et al., entitled "Method of Designing a Thermal Physical Vapor Deposition System". This conductance ratio promotes good pressure uniformity within manifold **60** and thereby minimizes flow non-uniformities through apertures **90** distributed along the length of the source despite potential local non-uniformities in vaporization rate.

One or more heat shields **70** are located adjacent the heated manifold **60** for the purpose of reducing the heat radiated to the facing target substrate. These heat shields are thermally connected to base block **20** for the purpose of drawing heat away from the shields. The upper portion of shields **70** is designed to lie below the plane of the apertures for the purpose of minimizing vapor condensation on their relatively cool surfaces.

Because only a small portion of organic material **10**, the portion resident in second heating region **35**, is heated to the rate-dependent vaporization temperature, while the bulk of the material is kept well below the vaporization temperature, it is possible to interrupt the vaporization by a way of interrupting heating in second heating region **35**. This can be done when a substrate surface is not being coated so as to conserve organic material **10** and minimize contamination of any associated apparatus, such as the walls of a deposition chamber, which will be described below.

Because permeable member **40** can be a fine mesh screen that prevents powder or compacted material from passing freely through it, vaporization apparatus **5** can be used in any orientation, i.e., it is an orientation independent apparatus. For example, vaporization apparatus **5** can be oriented

180° from what is shown in FIG. 1 so as to coat a substrate placed below it. This is an advantage not found in the heating boats of the prior art.

Although one preferred embodiment has been the use of vaporization apparatus 5 with a powder or compressed material that sublimes when heated, in some embodiments organic material 10 can be a material that liquefies before vaporization, and can be a liquid at the temperature of first heating region 25. In such a case, permeable member 40 can absorb and retain liquefied organic material 10 in a controllable manner via capillary action, thus permitting control of vaporization rate and providing orientation independence.

10 In practice, vaporization apparatus 5 can be used as follows. A quantity of organic material 10, which can comprise one or more components, is provided into chamber 15 of vaporization apparatus 5. In first heating region 25, organic material 10 is actively maintained below the vaporization temperature of each of its organic components. Second heating region 35 is heated to a temperature above the vaporization temperature of organic material 10 or each of the components thereof. Organic material 10 is metered at a controlled rate from first heating region 25 to second heating region 35. A thin cross-section of organic material 10 is heated at a desired rate-dependent vaporization temperature, whereby organic material 10 vaporizes and forms a film on a substrate surface. 15 When organic material 10 comprises multiple components, each component simultaneously vaporizes.

FIG. 3 shows a cross-sectional view of a second embodiment of a device of this disclosure. Vaporization apparatus 45 includes a piston 50, which in this embodiment is driven hydraulically by liquid 65. Vaporization apparatus 45 also includes first heating region 25, second heating region 35, base block 20, control passages 30, chamber 15, manifold 60, apertures 90, shields 70, and permeable member 40 as described above. Like vaporization apparatus 5, vaporization apparatus 45 can be adapted to the use of a liquid organic material 10. 25

While the use of two separated heating regions employing separate heating means in the orientation independent apparatus described above provides advantages with respect to preventing material degradation as discussed above, the invention may be employed with orientation independent apparatus including only
5 a single heating means sufficient to vaporize desired material (e.g., base block 20 of apparatus 5 may itself be heated to the material vaporization temperature). Referring to FIG. 4, e.g., a single heating region 36 is obtained in such embodiment.

Turning now to FIG. 5, there is shown an embodiment of a device
10 of this disclosure including a deposition chamber enclosing a substrate. Deposition chamber 80 is an enclosed apparatus that permits an OLED substrate 85 to be coated with organic material 10 transferred from vaporization apparatus 5. Deposition chamber 80 is held under controlled conditions, e.g. a pressure of 1 Torr or less provided by vacuum source 100. Deposition chamber 80 includes
15 load lock 75 which can be used to load uncoated OLED substrates 85, and unload coated OLED substrates. OLED substrate 85 can be moved by translational apparatus 95 to provide even coating of vaporized organic material 10 over the entire surface of OLED substrate 85. Although vaporization apparatus is shown as partially enclosed by deposition chamber 80, it will be understood that other
20 arrangements are possible, including arrangements wherein vaporization apparatus 5 is entirely enclosed by deposition chamber 80.

In practice, an OLED substrate 85 is placed in deposition chamber 80 via load lock 75 and held by translational apparatus 95 or associated apparatus. Vaporization apparatus 5 is operated as described above, and translational
25 apparatus 95 moves OLED substrate 85 perpendicular to the direction of emission of organic material 10 vapors from vaporization apparatus 5, thus forming a film of organic material 10 on the surface of OLED substrate 85.

Referring to Fig. 7, a system for the deposition of vaporized materials on a substrate comprises at least first and second orientation-independent
30 apparatuses 5 for directing vaporized organic materials onto a flexible substrate

200 to form first and second films, each of the first and second orientation-independent apparatuses being arranged in a different relative orientation to provide consistent deposition regardless of orientation. In the embodiment shown in Fig. 7, a flexible substrate 200 is wound around feed roller 202, deposition
5 roller 204, and take-up roller 206. Positioning and tension control rollers 208 provide control of the substrate movement. If desired, the flexible substrate can be cut into sheets after deposition (not shown). A plurality of vaporization apparatuses 5 are located around the deposition roller 204 at a variety of orientations to deposit vaporized materials onto the flexible substrate 200. The
10 entire assembly may be provided within an vacuum chamber 212 with access hatches 210. In operation, the take-up and feed rollers 206 and 202 together with the control rollers 208 move the flexible substrate 200 past the vaporization apparatus 5 to deposit thin films of materials onto the flexible substrate 200.

Because identical vaporization apparatuses 5 are used, consistent
15 control of the devices and deposition process is more readily achieved. Moreover, costs are reduced by using a single type of apparatus rather than a plurality of unique apparatuses. In prior-art designs requiring deposition from multiple orientations, deposition devices having unique chimneys, heating geometries, or other unique attributes are necessary, thereby causing difficulties in consistent
20 control and manufacturing process.

In an alternative embodiment, illustrated in Fig. 8, evaporated materials are deposited on a substrate from above and from the side. Referring to Fig. 8, belt rollers 209 transport a belt 211 that provides a surface on which a plurality of substrates 85 may be affixed and that travel in sequence beneath
25 vaporization apparatuses 5 to sequentially deposit thin films of materials from above on the substrate. At a later point in the process, materials may be deposited onto a vertical substrate from the side. Alternatively, control rollers could control a flexible substrate (not shown) traveling past vaporization apparatuses 5 rather than substrates affixed to a belt. Further, while not depicted, apparatuses 5 may be

located to deposit materials on each side of a substrate to create, e.g. a display or illumination device that can emit light from both sides of the substrate, or provide filter or protective layers on either side.

Any of the above configurations may be used with masks. The masks may be affixed to a substrate or to a vaporization apparatus. Referring to Fig. 9, a vaporization apparatus **5** evaporates material through a mask **87** onto a substrate **85**.

In the configurations of Figs. 8 and 9, a flexible substrate may be held flat by an underlying, rigid support. Referring to Fig. **10**, a vaporization apparatus **5** evaporates material through a mask **87** onto a flexible substrate **200**. Beneath the flexible substrate **200** is located a rigid, flat support **220**.

The present invention may be employed in a variety of configurations. For example, the present invention may be employed with vaporization and deposition apparatus **5** in a linear source configuration wherein the apparatus is configured to provide a vapor plume along a line. This can be accomplished by constructing the apparatus in a rectangular structure having a large aspect ratio. Referring to Fig. 11, an embodiment of the apparatus in a linear source is illustrated with aperture **90**, piston **50**, permeable member **40** and chamber **15**. Alternatively, referring to Fig. 12, an embodiment of the apparatus in a point source is illustrated with aperture **90**, piston **50**, permeable member **40** and chamber **15**. Referring to Fig. 13, in another embodiment, a planar source apparatus having aperture **90**, piston **50**, permeable member **40** and chamber **15** is illustrated. Because the vaporization apparatus of the present invention is orientation-independent, it may be employed on a moving platform moving in any direction or dimension. In particular, it is known to move point sources in rotating patterns; the present invention provides an improved deposition device in such an application.

Turning now to FIG. 6, there is shown a cross-sectional view of a pixel of a light-emitting OLED device **110** that can be prepared in part according to the present invention. The OLED device **110** includes at a minimum a substrate

120, a cathode 190, an anode 130 spaced from cathode 190, and a light-emitting layer 150. The OLED device can also include a hole-injecting layer 135, a hole-transporting layer 140, an electron-transporting layer 155, and an electron-injecting layer 160. Hole-injecting layer 135, hole-transporting layer 140, light-emitting layer 150, electron-transporting layer 155, and electron-injecting layer 160 comprise a series of organic layers 170 disposed between anode 130 and cathode 190. Organic layers 170 are the layers most desirably deposited by the device and method of this invention. These components will be described in more detail.

10 Substrate 120 can be an organic solid, an inorganic solid, or include organic and inorganic solids. Substrate 120 can be rigid or flexible and can be processed as separate individual pieces, such as sheets or wafers, or as a continuous roll. Typical substrate materials include glass, plastic, metal, ceramic, semiconductor, metal oxide, semiconductor oxide, semiconductor nitride, or combinations thereof. The substrate may be a thin, flexible foil, for example of plastic or metal. Substrate 120 can be a homogeneous mixture of materials, a composite of materials, or multiple layers of materials. Substrate 120 can be an OLED substrate, that is a substrate commonly used for preparing OLED devices, e.g. active-matrix low-temperature polysilicon or amorphous-silicon TFT

15 substrate. The substrate 120 can either be light transmissive or opaque, depending on the intended direction of light emission. The light transmissive property is desirable for viewing the EL emission through the substrate. Transparent glass or plastic are commonly employed in such cases. For applications where the EL emission is viewed through the top electrode, the transmissive characteristic of the

20 bottom support is immaterial, and therefore can be light transmissive, light absorbing or light reflective. Substrates for use in this case include, but are not limited to, glass, plastic, semiconductor materials, ceramics, and circuit board materials, or any others commonly used in the formation of OLED devices, which

25 can be either passive-matrix devices or active-matrix devices.

An electrode is formed over substrate **120** and is most commonly configured as an anode **130**. When EL emission is viewed through the substrate **120**, anode **130** should be transparent or substantially transparent to the emission of interest. Common transparent anode materials useful in this invention are

5 indium-tin oxide and tin oxide, but other metal oxides can work including, but not limited to, aluminum- or indium-doped zinc oxide, magnesium-indium oxide, and nickel-tungsten oxide. In addition to these oxides, metal nitrides such as gallium nitride, metal selenides such as zinc selenide, and metal sulfides such as zinc sulfide, can be used as an anode material. For applications where EL emission is

10 viewed through the top electrode, the transmissive characteristics of the anode material are immaterial and any conductive material can be used, transparent, opaque or reflective. Example conductors for this application include, but are not limited to, gold, iridium, molybdenum, palladium, and platinum. The preferred anode materials, transmissive or otherwise, have a work function of 4.1 eV or

15 greater. Desired anode materials can be deposited by any suitable way such as evaporation, sputtering, chemical vapor deposition, or electrochemical means. Anode materials can be patterned using well known photolithographic processes.

While not always necessary, it is often useful that a hole-injecting layer **135** be formed over anode **130** in an organic light-emitting display. The hole-

20 injecting material can serve to improve the film formation property of subsequent organic layers and to facilitate injection of holes into the hole-transporting layer. Suitable materials for use in hole-injecting layer **135** include, but are not limited to, porphyrinic compounds as described in U.S. Patent 4,720,432, plasma-deposited fluorocarbon polymers as described in U.S. Patent 6,208,075, and

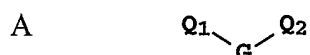
25 inorganic oxides including vanadium oxide (VOx), molybdenum oxide (MoOx), nickel oxide (NiOx), etc. Alternative hole-injecting materials reportedly useful in organic EL devices are described in EP 0 891 121 A1 and EP 1 029 909 A1.

While not always necessary, it is often useful that a hole-transporting layer **140** be formed and disposed over anode **130**. Desired hole-

30 transporting materials can be deposited by any suitable way such as evaporation,

sputtering, chemical vapor deposition, electrochemical means, thermal transfer, or laser thermal transfer from a donor material, and can be deposited by the device and method described herein. Hole-transporting materials useful in hole-transporting layer 140 are well known to include compounds such as an aromatic tertiary amine, where the latter is understood to be a compound containing at least one trivalent nitrogen atom that is bonded only to carbon atoms, at least one of which is a member of an aromatic ring. In one form the aromatic tertiary amine can be an arylamine, such as a monoarylamine, diarylamine, triarylamine, or a polymeric arylamine. Exemplary monomeric triarylamine are illustrated by Klupfel et al. in U.S. Patent 3,180,730. Other suitable triarylamine substituted with one or more vinyl radicals and/or comprising at least one active hydrogen-containing group are disclosed by Brantley et al. in U.S. Patents 3,567,450 and 3,658,520.

A more preferred class of aromatic tertiary amines are those which include at least two aromatic tertiary amine moieties as described in U.S. Patents 4,720,432 and 5,061,569. Such compounds include those represented by structural Formula A

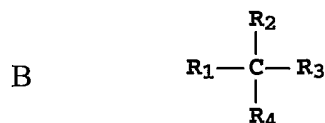


wherein:

Q₁ and Q₂ are independently selected aromatic tertiary amine moieties; and G is a linking group such as an arylene, cycloalkylene, or alkylene group of a carbon to carbon bond.

In one embodiment, at least one of Q₁ or Q₂ contains a polycyclic fused ring structure, e.g., a naphthalene. When G is an aryl group, it is conveniently a phenylene, biphenylene, or naphthalene moiety.

A useful class of triarylamine satisfying structural Formula A and containing two triarylamine moieties is represented by structural Formula B



where:

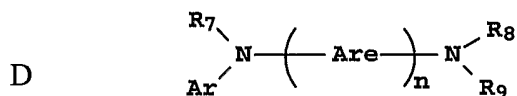
R₁ and R₂ each independently represent a hydrogen atom, an aryl group, or an alkyl group or R₁ and R₂ together represent the atoms completing a cycloalkyl group; and

R₃ and R₄ each independently represent an aryl group, which is in turn substituted with a diaryl substituted amino group, as indicated by structural Formula C



wherein R₅ and R₆ are independently selected aryl groups. In one embodiment, at least one of R₅ or R₆ contains a polycyclic fused ring structure, e.g., a naphthalene.

Another class of aromatic tertiary amines are the tetraaryldiamines. Desirable tetraaryldiamines include two diarylamino groups, such as indicated by Formula C, linked through an arylene group. Useful tetraaryldiamines include those represented by Formula D



wherein:

each Are is an independently selected arylene group, such as a phenylene or anthracene moiety;

n is an integer of from 1 to 4; and

Ar, R₇, R₈, and R₉ are independently selected aryl groups.

In a typical embodiment, at least one of Ar, R₇, R₈, and R₉ is a polycyclic fused ring structure, e.g., a naphthalene.

The various alkyl, alkylene, aryl, and arylene moieties of the foregoing structural Formulae A, B, C, D, can each in turn be substituted. Typical substituents include alkyl groups, alkoxy groups, aryl groups, aryloxy groups, and halogens such as fluoride, chloride, and bromide. The various alkyl and alkylene
5 moieties typically contain from 1 to about 6 carbon atoms. The cycloalkyl moieties can contain from 3 to about 10 carbon atoms, but typically contain five, six, or seven carbon atoms--e.g., cyclopentyl, cyclohexyl, and cycloheptyl ring structures. The aryl and arylene moieties are usually phenyl and phenylene moieties.

The hole-transporting layer in an OLED device can be formed of a
10 single or a mixture of aromatic tertiary amine compounds. Specifically, one can employ a triarylamine, such as a triarylamine satisfying the Formula B, in combination with a tetraaryldiamine, such as indicated by Formula D. When a triarylamine is employed in combination with a tetraaryldiamine, the latter is positioned as a layer interposed between the triarylamine and the electron-
15 injecting and transporting layer. The device and method described herein can be used to deposit single- or multi-component layers, and can be used to sequentially deposit multiple layers.

Another class of useful hole-transporting materials includes polycyclic aromatic compounds as described in EP 1 009 041. In addition,
20 polymeric hole-transporting materials can be used such as poly(N-vinylcarbazole) (PVK), polythiophenes, polypyrrole, polyaniline, and copolymers such as poly(3,4-ethylenedioxythiophene)/poly(4-styrenesulfonate) also called PEDOT/PSS.

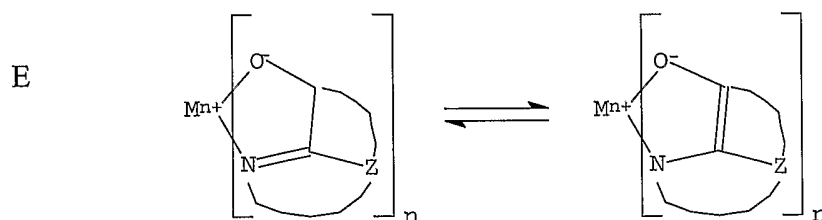
Light-emitting layer **150** produces light in response to hole-electron
25 recombination. Light-emitting layer **150** is commonly disposed over hole-transporting layer **140**. Desired organic light-emitting materials can be deposited by any suitable way such as evaporation, sputtering, chemical vapor deposition, electrochemical means, or radiation thermal transfer from a donor material, and can be deposited by the device and method described herein. Useful organic light-
30 emitting materials are well known. As more fully described in U.S. Patents

4,769,292 and 5,935,721, the light-emitting layers of the organic EL element comprise a luminescent or fluorescent material where electroluminescence is produced as a result of electron-hole pair recombination in this region. The light-emitting layers can be comprised of a single material, but more commonly include a host material doped with a guest compound or dopant where light emission comes primarily from the dopant. The dopant is selected to produce color light having a particular spectrum. The host materials in the light-emitting layers can be an electron-transporting material, as defined below, a hole-transporting material, as defined above, or another material that supports hole-electron recombination.

The dopant is usually chosen from highly fluorescent dyes, but phosphorescent compounds, e.g., transition metal complexes as described in WO 98/55561, WO 00/18851, WO 00/57676, and WO 00/70655 are also useful. Dopants are typically coated as 0.01 to 10 % by weight into the host material. The device and method described herein can be used to coat multi-component guest/host layers without the need for multiple vaporization sources.

Host and emitting molecules known to be of use include, but are not limited to, those disclosed in U.S. Patents 4,768,292; 5,141,671; 5,150,006; 5,151,629; 5,294,870; 5,405,709; 5,484,922; 5,593,788; 5,645,948; 5,683,823; 5,755,999; 5,928,802; 5,935,720; 5,935,721; and 6,020,078.

Metal complexes of 8-hydroxyquinoline and similar derivatives (Formula E) constitute one class of useful host materials capable of supporting electroluminescence, and are particularly suitable for light emission of wavelengths longer than 500 nm, e.g., green, yellow, orange, and red.



wherein:

M represents a metal;

n is an integer of from 1 to 3; and

Z independently in each occurrence represents the atoms completing a nucleus having at least two fused aromatic rings.

From the foregoing it is apparent that the metal can be a
5 monovalent, divalent, or trivalent metal. The metal can, for example, be an alkali metal, such as lithium, sodium, or potassium; an alkaline earth metal, such as magnesium or calcium; or an earth metal, such as boron or aluminum. Generally any monovalent, divalent, or trivalent metal known to be a useful chelating metal can be employed.

10 Z completes a heterocyclic nucleus containing at least two fused aromatic rings, at least one of which is an azole or azine ring. Additional rings, including both aliphatic and aromatic rings, can be fused with the two required rings, if required. To avoid adding molecular bulk without improving on function the number of ring atoms is usually maintained at 18 or less.

15 The host material in light-emitting layer **150** can be an anthracene derivative having hydrocarbon or substituted hydrocarbon substituents at the 9 and 10 positions. For example, derivatives of 9,10-di-(2-naphthyl)anthracene constitute one class of useful host materials capable of supporting electroluminescence, and are particularly suitable for light emission of
20 wavelengths longer than 400 nm, e.g., blue, green, yellow, orange or red.

Benzazole derivatives constitute another class of useful host materials capable of supporting electroluminescence, and are particularly suitable for light emission of wavelengths longer than 400 nm, e.g., blue, green, yellow, orange or red. An example of a useful benzazole is 2, 2', 2''-(1,3,5-
25 phenylene)tris[1-phenyl-1H-benzimidazole].

Desirable fluorescent dopants include perylene or derivatives of perylene, derivatives of anthracene, tetracene, xanthene, rubrene, coumarin, rhodamine, quinacridone, dicyanomethylenepyran compounds, thiopyran compounds, polymethine compounds, pyrilium and thiapyrilium compounds,
30 derivatives of distyrylbenzene or distyrylbiphenyl, bis(azinyl)methane boron complex compounds, and carbostyryl compounds.

Other organic emissive materials can be polymeric substances, e.g. polyphenylenevinylene derivatives, dialkoxy-polyphenylenevinlenes, poly-para-phenylene derivatives, and polyfluorene derivatives, as taught by Wolk et al. in commonly assigned U.S. Patent 6,194,119 B1 and references cited therein.

5 While not always necessary, it is often useful that OLED device **110** includes an electron-transporting layer **155** disposed over light-emitting layer **150**. Desired electron-transporting materials can be deposited by any suitable way such as evaporation, sputtering, chemical vapor deposition, electrochemical means, thermal transfer, or laser thermal transfer from a donor material, and can
10 be deposited by the device and method described herein. Preferred electron-transporting materials for use in electron-transporting layer **155** are metal chelated oxinoid compounds, including chelates of oxine itself (also commonly referred to as 8-quinolinol or 8-hydroxyquinoline). Such compounds help to inject and transport electrons and exhibit both high levels of performance and are readily
15 fabricated in the form of thin films. Exemplary of contemplated oxinoid compounds are those satisfying structural Formula E, previously described.

Other electron-transporting materials include various butadiene derivatives as disclosed in U.S. Patent 4,356,429 and various heterocyclic optical brighteners as described in U.S. Patent 4,539,507. Benzazoles satisfying structural
20 Formula G are also useful electron-transporting materials.

Other electron-transporting materials can be polymeric substances, e.g. polyphenylenevinylene derivatives, poly-para-phenylene derivatives, polyfluorene derivatives, polythiophenes, polyacetylenes, and other conductive polymeric organic materials such as those listed in *Handbook of Conductive*
25 *Molecules and Polymers*, Vols. 1-4, H.S. Nalwa, ed., John Wiley and Sons, Chichester (1997).

An electron-injecting layer **160** can also be present between the cathode and the electron-transporting layer. Examples of electron-injecting materials include alkaline or alkaline earth metals, alkali halide salts, such as LiF
30 mentioned above, or alkaline or alkaline earth metal doped organic layers.

Cathode **190** is formed over the electron-transporting layer **155** or over light-emitting layer **150** if an electron-transporting layer is not used. When light emission is through the anode **130**, the cathode material can be comprised of nearly any conductive material. Desirable materials have good film-forming
5 properties to ensure good contact with the underlying organic layer, promote electron injection at low voltage, and have good stability. Useful cathode materials often contain a low work function metal (< 3.0 eV) or metal alloy. One preferred cathode material is comprised of a Mg:Ag alloy wherein the percentage of silver is in the range of 1 to 20 %, as described in U.S. Patent 4,885,221. Another suitable
10 class of cathode materials includes bilayers comprised of a thin layer of a low work function metal or metal salt capped with a thicker layer of conductive metal. One such cathode is comprised of a thin layer of LiF followed by a thicker layer of Al as described in U.S. Patent 5,677,572. Other useful cathode materials include, but are not limited to, those disclosed in U.S. Patents 5,059,861; 5,059,862; and
15 6,140,763.

When light emission is viewed through cathode **190**, it must be transparent or nearly transparent. For such applications, metals must be thin or one must use transparent conductive oxides, or include these materials. Optically transparent cathodes have been described in more detail in U.S. Patent 5,776,623.
20 Cathode materials can be deposited by evaporation, sputtering, or chemical vapor deposition. When needed, patterning can be achieved through many well known methods including, but not limited to, through-mask deposition, integral shadow masking as described in U.S. Patent 5,276,380 and EP 0 732 868, laser ablation, and selective chemical vapor deposition.

25 Cathode materials can be deposited by evaporation, sputtering, or chemical vapor deposition. When needed, patterning can be achieved through many well known methods including, but not limited to, through-mask deposition, integral shadow masking as described in U.S. Patent 5,276,380 and EP 0 732 868, laser ablation, and selective chemical vapor deposition.

PARTS LIST

5	vaporization apparatus
10	organic material
15	chamber
20	base block
36	single heating region
25	first heating region
30	control passage
35	second heating region
40	permeable member
45	vaporization apparatus
50	piston
60	manifold
65	liquid
70	shield
75	load lock
80	deposition chamber
85	OLED substrate
87	mask
90	aperture
95	translational apparatus
100	vacuum source
110	OLED device
120	substrate
130	anode
135	hole-injecting layer
140	hole-transporting layer
150	light-emitting layer
155	electron-transporting layer

PARTS LIST (con't)

160	electron-injecting layer
170	organic layers
190	cathode
200	flexible substrate
202	feed roller
204	deposition roller
206	take-up roller
208	control roller
209	belt roller
210	access hatch
211	belt
212	vacuum chamber
220	rigid planar support

CLAIMS:

1. A system for the deposition of vaporized materials on a substrate, comprising at least first and second orientation-independent apparatuses for directing vaporized organic materials onto a substrate surface to form first and second films, each of the first and second orientation-independent apparatuses
5 being arranged in a different relative orientation and comprising:
- a) a chamber containing a quantity of material;
 - b) a permeable member at one end of the chamber with a heating element for vaporizing the material; and
 - 10 c) a piston at the other end of the chamber for continuously feeding the material toward the permeable member as it is vaporized, whereby organic material vaporizes at a desired rate-dependent vaporization temperature at the one end of the chamber.
2. The system claimed in claim 1 wherein the substrate is flexible.
- 15 3. The system claimed in claim 2 wherein the substrate is wound around a roller, and the first and second vaporization apparatuses are located around the roller at a variety of orientations to deposit vaporized materials onto the flexible substrate.
4. The system claimed in claim 1 wherein the substrate is
20 horizontal.
5. The system claimed in claim 4 wherein material is deposited by at least one orientation-independent apparatus from below the substrate.
6. The system claimed in claim 4 wherein material is deposited by at least one orientation-independent apparatus from above the substrate.
- 25 7. The system claimed in claim 4 wherein material is deposited by at least one orientation-independent apparatus from above the substrate, and by at least one orientation-independent apparatus from below the substrate.

8. The system claimed in claim 1 wherein the substrate is vertical.
9. The system according to claim 1 further including a deposition chamber enclosing the substrate and the orientation-independent apparatuses.
10. The system claimed in claim 1 wherein the piston is a
5 mechanical piston.
11. The system claimed in claim 1 wherein the piston is a hydraulic piston.
12. The system claimed in claim 1 wherein the orientation-independent apparatus is a point source deposition apparatus.
- 10 13. The system claimed in claim 1 wherein the orientation-independent apparatus is a linear source deposition apparatus.
14. The system claimed in claim 1 wherein the orientation-independent apparatus is a planar source deposition apparatus.
15. A method of depositing thin-films on a substrate comprising
15 the steps of: a) providing a substrate; b) providing at least first and second orientation-independent material vaporization and deposition apparatuses; c) continuously moving the substrate past the first and second orientation-independent apparatuses; and d) directing vaporized organic materials in distinct relative directions from each of the first and second orientation-independent
20 apparatuses and coating thin films of vaporized material on the substrate.
16. A method as claimed in claim 15, wherein each orientation-independent apparatus comprises:
- a) a chamber containing a quantity of material;

b) a permeable member at one end of the chamber with a heating element for vaporizing the material; and

c) a piston at the other end of the chamber for continuously feeding the material toward the permeable member as it is vaporized, whereby organic material vaporizes at a desired rate-dependent vaporization temperature at the one end of the chamber.

17. The method of depositing thin-films on a substrate claimed in claim 15 wherein the substrate is continuous.

18. The method of depositing thin-films on a substrate claimed in claim 15 wherein the substrate is flexible.

19. The method of depositing thin-films on a substrate claimed in claim 15 wherein the substrate is rigid and is positioned on a traveling belt to move the substrate.

20. The method of depositing thin-films on a substrate claimed in claim 15 wherein the vaporized materials are OLED materials.

21. The method claimed in claim 15 wherein the orientation-independent apparatus is a point source deposition apparatus.

22. The orientation-independent apparatus claimed in claim 15 wherein the orientation-independent apparatus is a linear source deposition apparatus.

23. The orientation-independent apparatus claimed in claim 15 wherein the orientation-independent apparatus is a planar source deposition apparatus.

24. An orientation-independent apparatus for vaporizing and depositing organic materials onto a substrate surface to form a film, comprising:

- a) a chamber containing a quantity of material;
- b) a permeable member at one end of the chamber with a single heating element for vaporizing the material at a desired rate-dependent vaporization temperature at the one end of the chamber; and
- 5 c) a piston at the other end of the chamber for continuously feeding the material toward the permeable member as it is vaporized.

25. The orientation-independent apparatus claimed in claim 24 wherein the piston is a mechanical piston.

10 26. The orientation-independent apparatus claimed in claim 24 wherein the piston is a hydraulic piston.

27. The orientation-independent apparatus claimed in claim 24 wherein the apparatus is a point source deposition apparatus.

28. The orientation-independent apparatus claimed in claim 24 wherein the apparatus is a linear source deposition apparatus.

15 29. The orientation-independent apparatus claimed in claim 24 wherein the apparatus is a planar source deposition apparatus.

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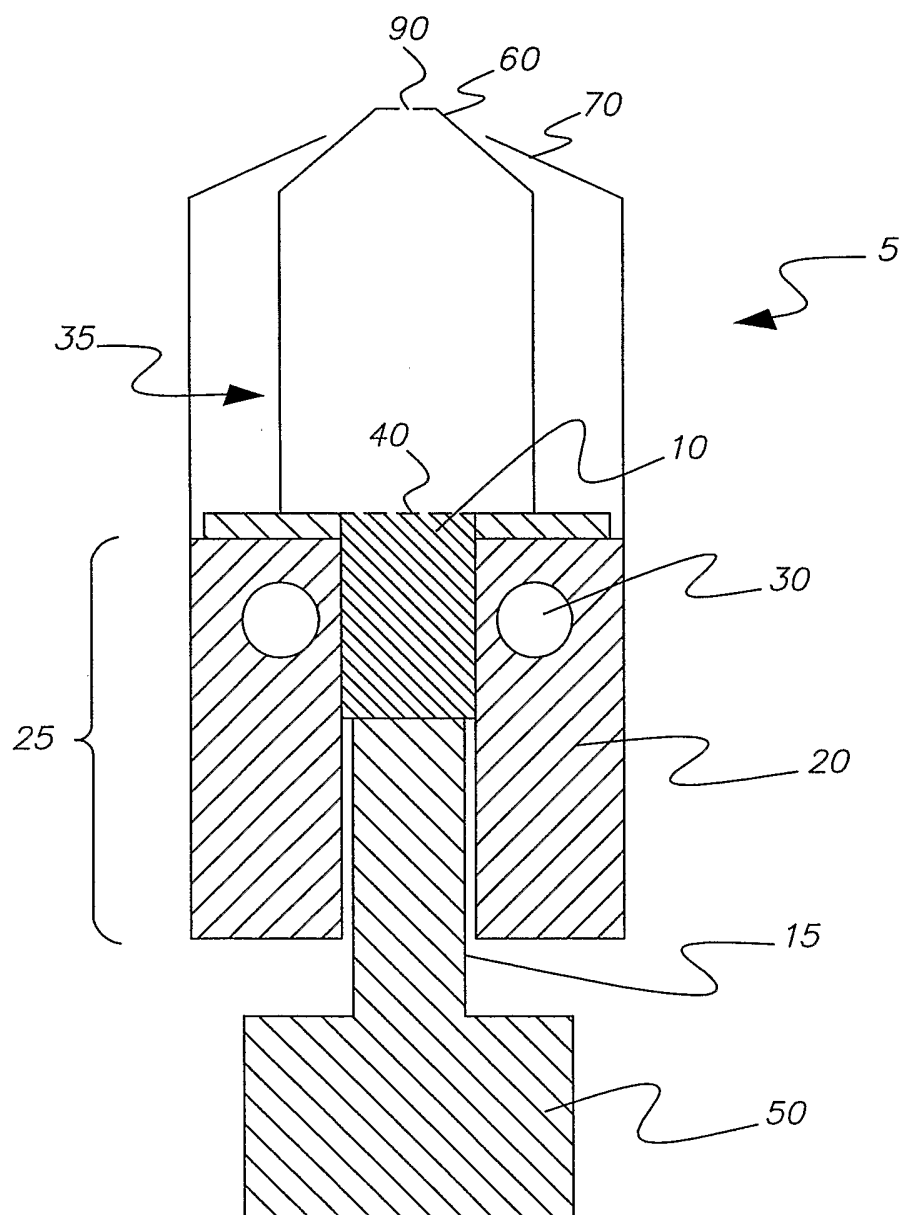


FIG. 1

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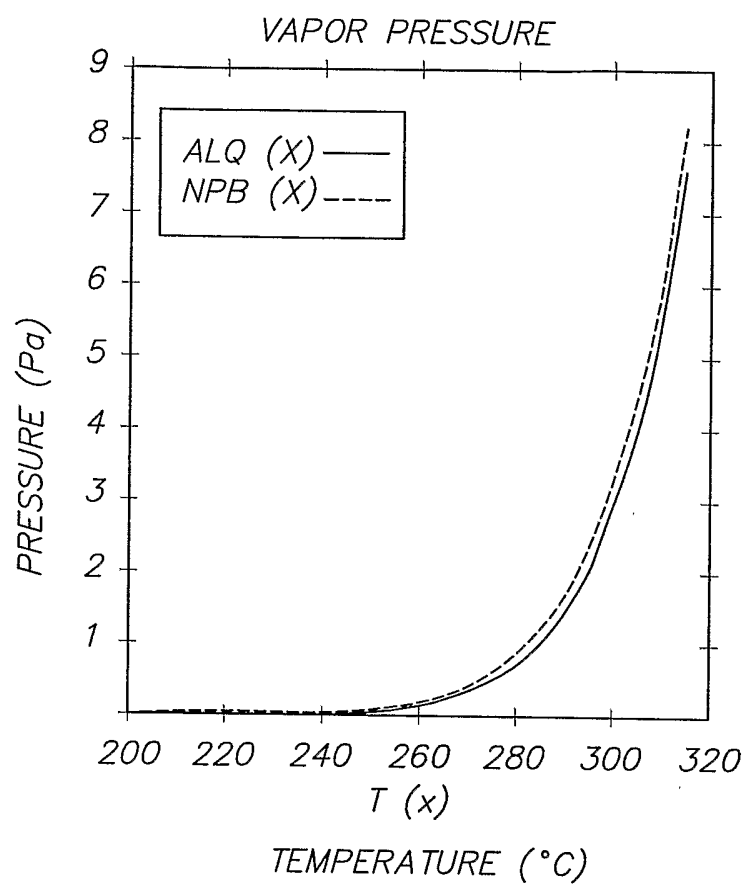


FIG. 2

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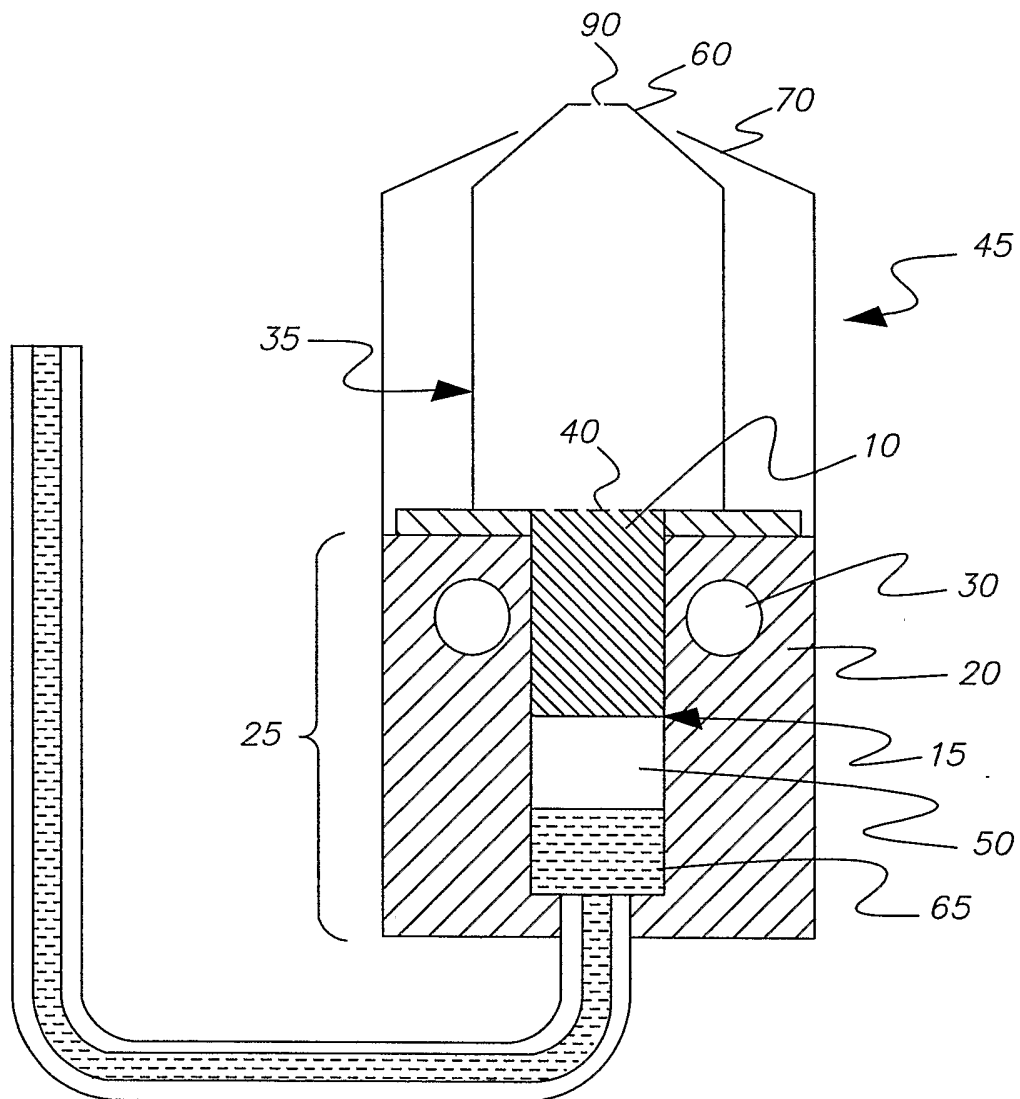


FIG. 3

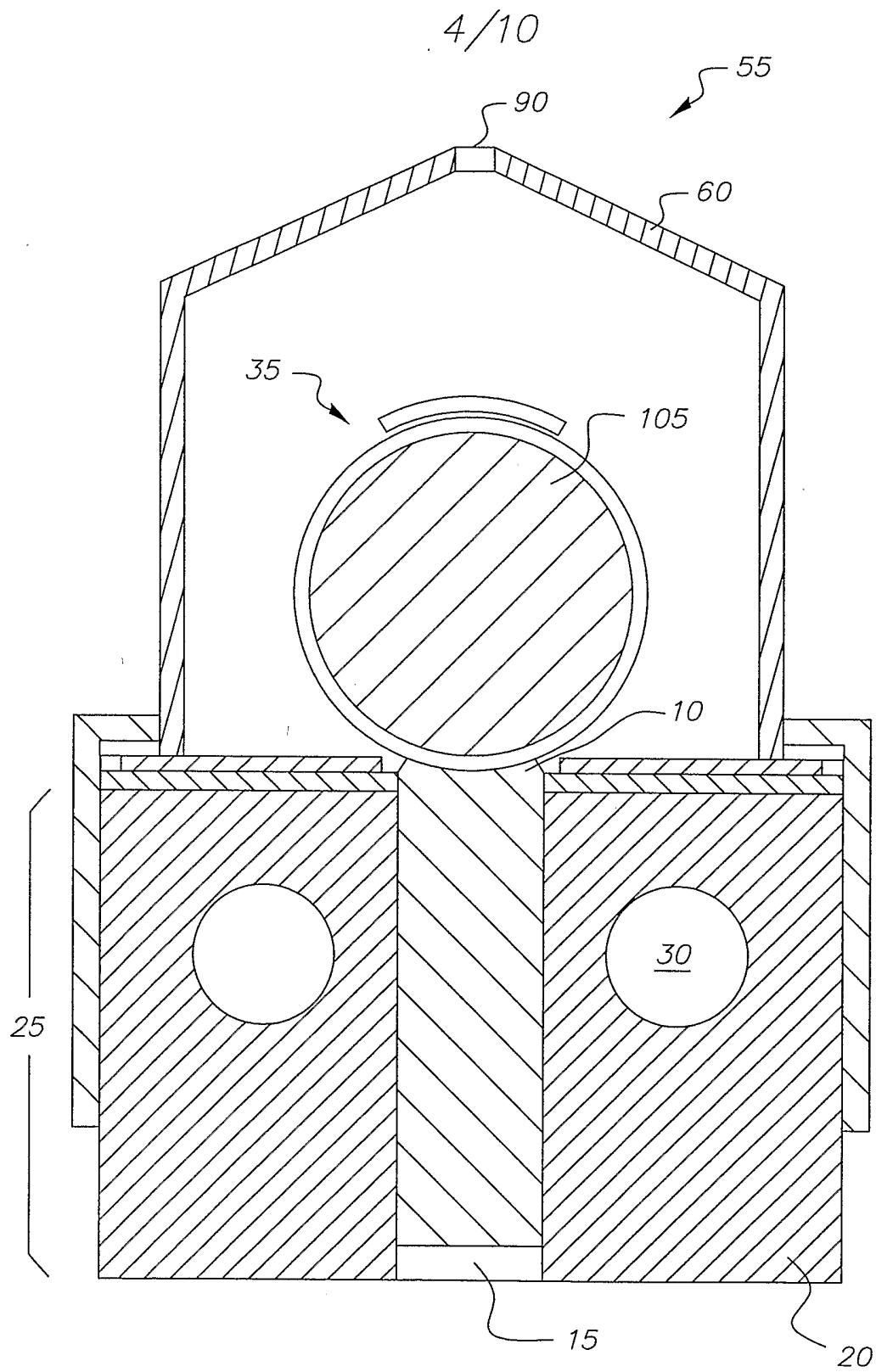


FIG. 4

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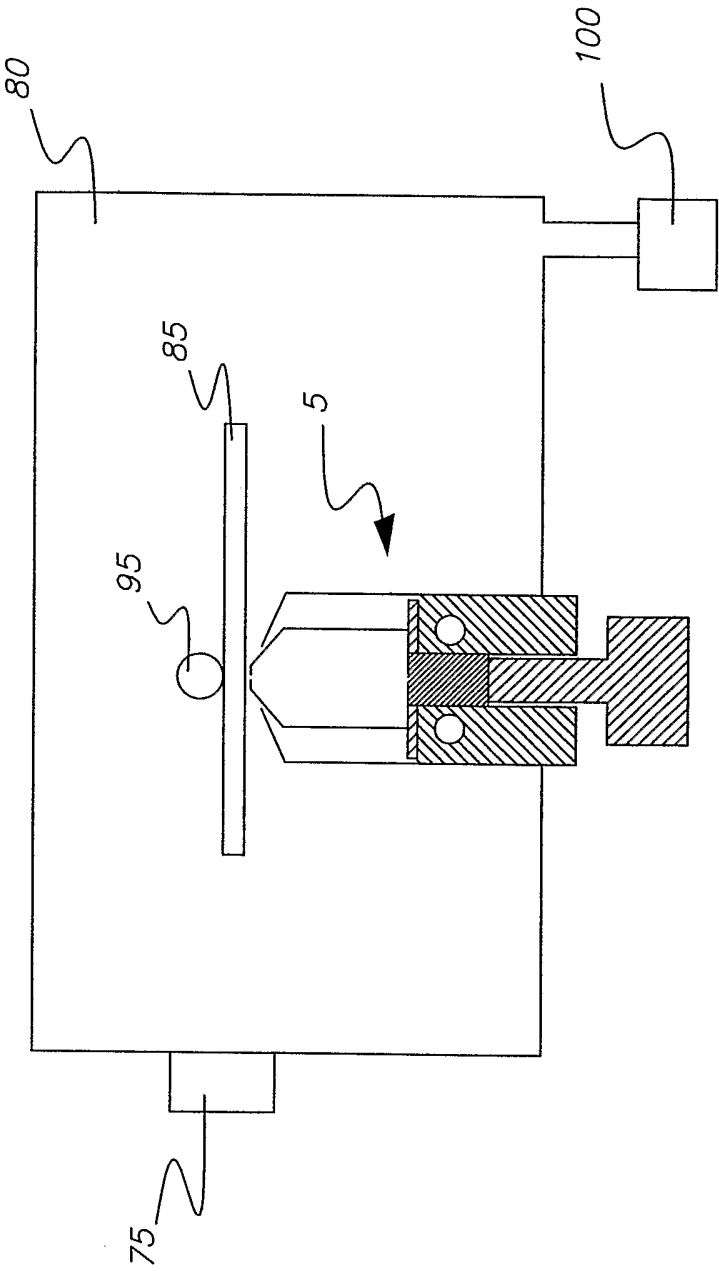


FIG. 5

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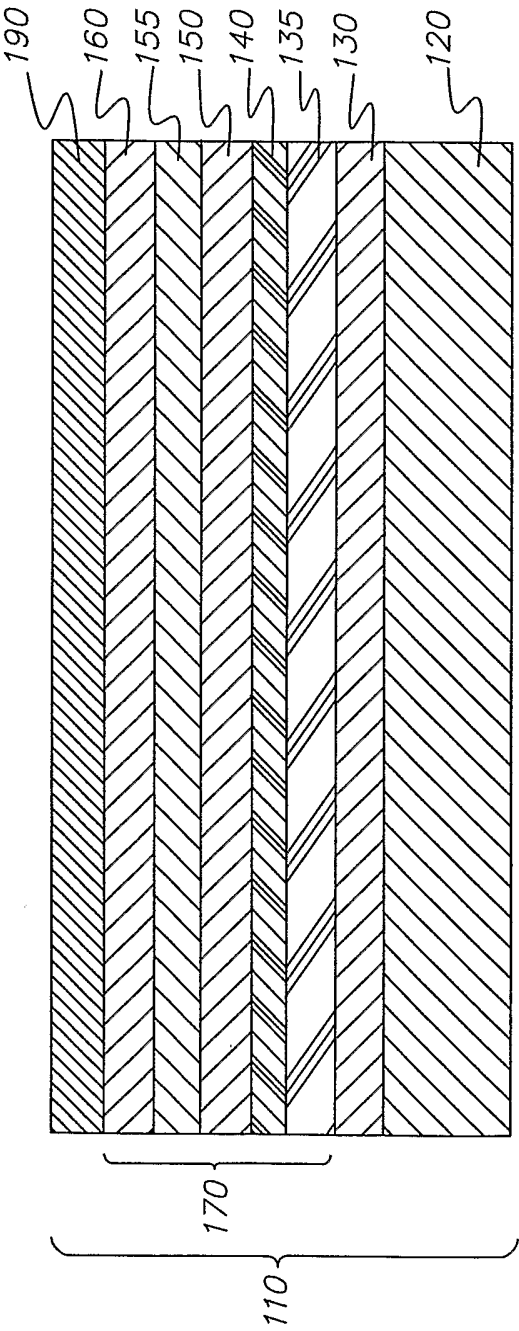


FIG. 6

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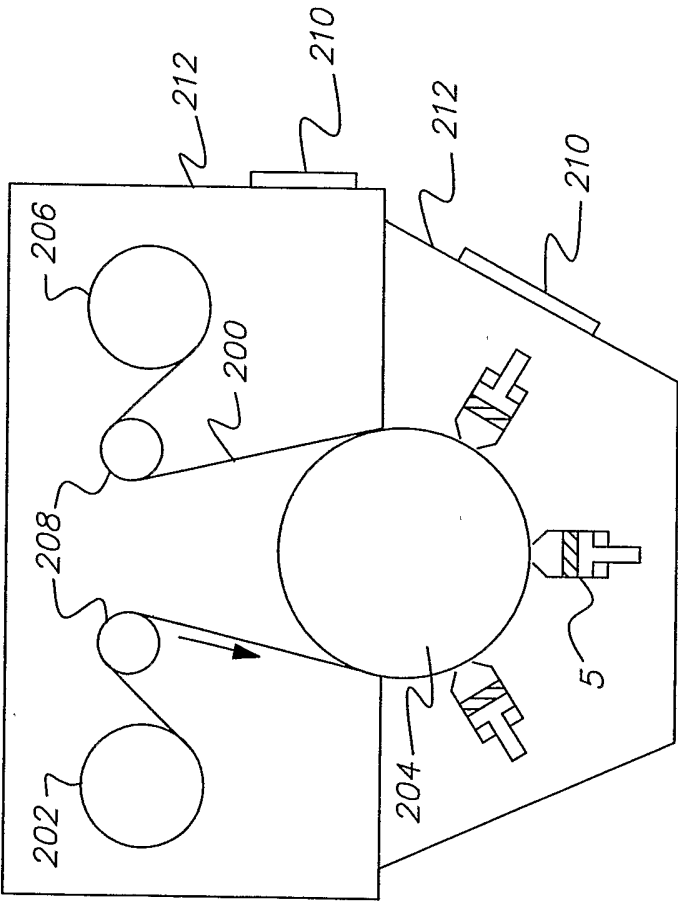


FIG. 7

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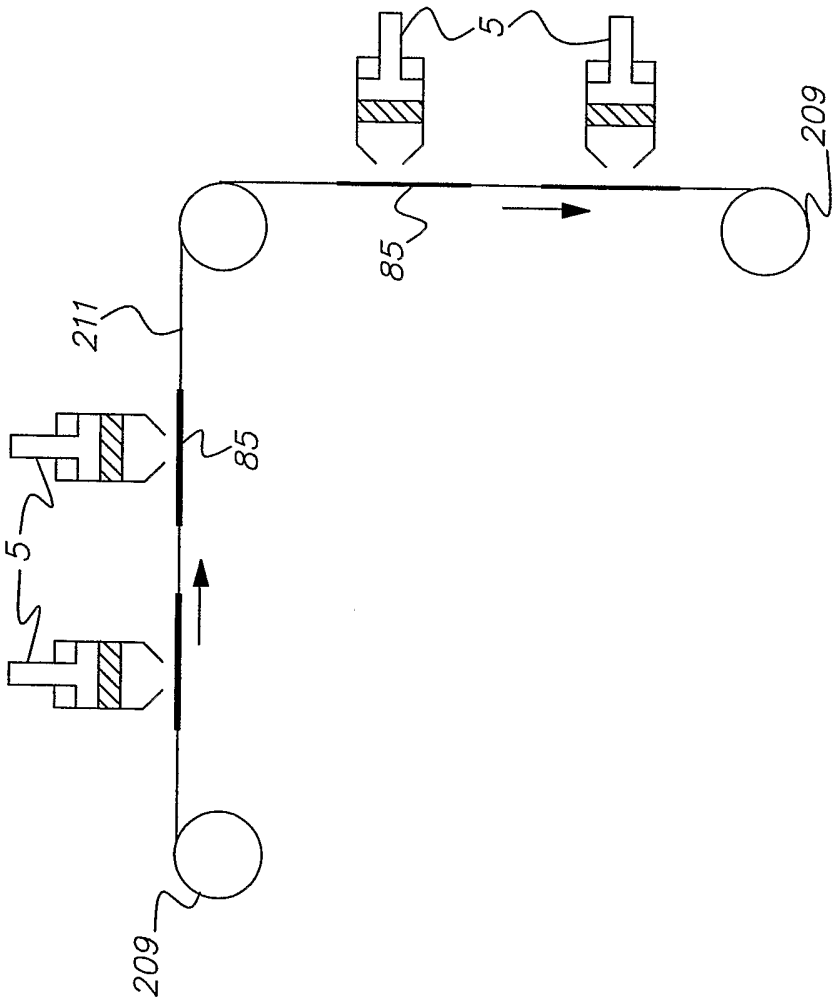


FIG. 8

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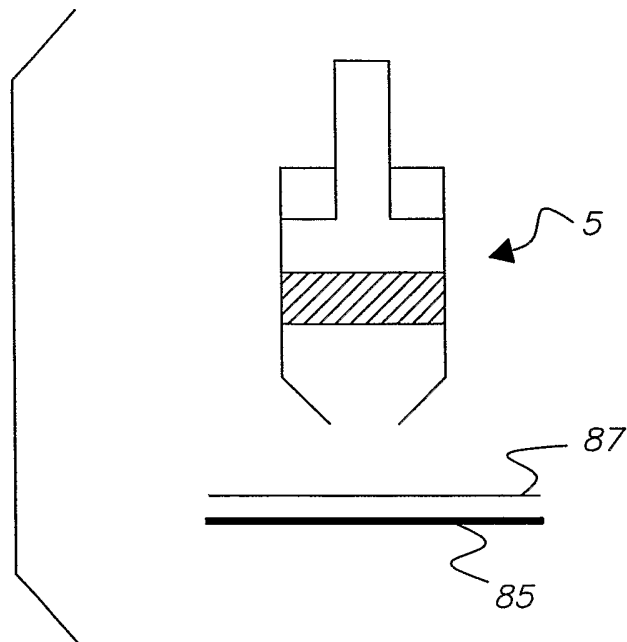


FIG. 9

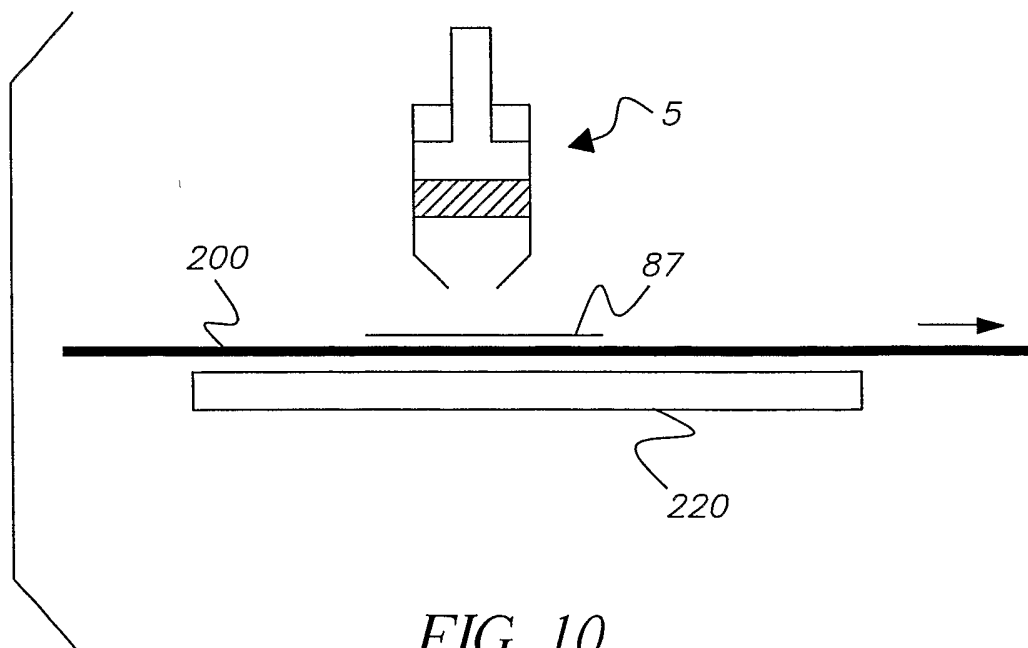


FIG. 10

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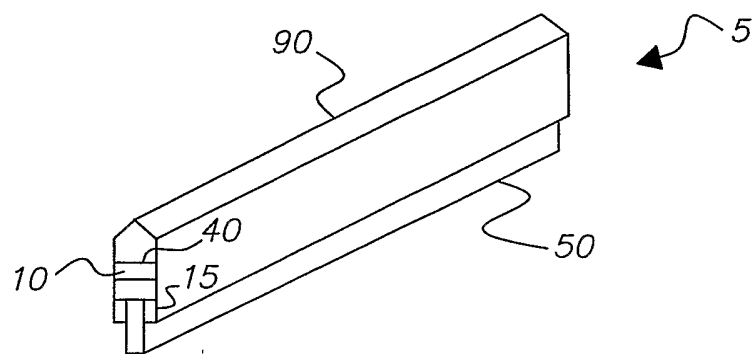


FIG. 11

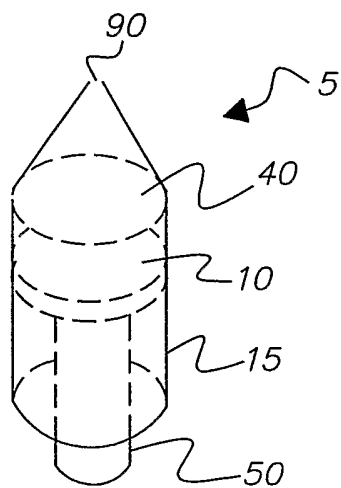


FIG. 12

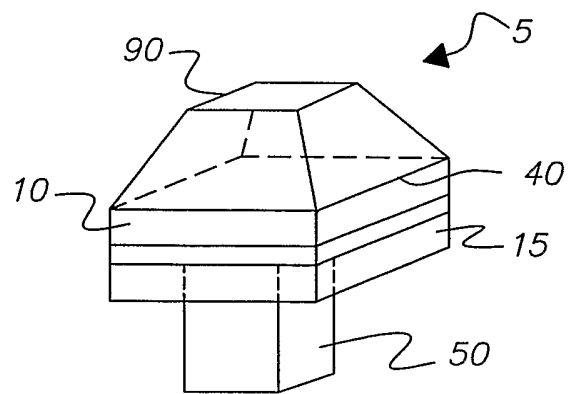


FIG. 13

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US2005/014888

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 H05B33/10 C23C14/24

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 H05B C23C H01L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

- *A* document defining the general state of the art which is not considered to be of particular relevance
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- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
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Date of the actual completion of the international search

18 July 2005

Date of mailing of the international search report

26/07/2005

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Vanier, C

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US2005/014888

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2001/022272 A1 (PLESTER GEORGE ET AL) 20 September 2001 (2001-09-20) Apparatus es and methods for use in vacuum vapor deposition coating. Continuous feeding. Separate melting and evaporating crucibles. Mainly designed for silicon coatings.claims 1-5 figures 1,2a paragraphs '0035! - '0037!	1-29
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A	EP 1 176 647 A (EASTMAN KODAK COMPANY) 30 January 2002 (2002-01-30) In vacuum vapor deposition apparatus and method for making electrode in OLED. Continuous feeding of the alloy through a wire of mat. to be evaporated. figure 3 paragraphs '0009!, '0023!	1-29
A	EP 1 391 944 A (EASTMAN KODAK COMPANY) 25 February 2004 (2004-02-25) Apparatus and method of depositing layer of organic electrolum. mat. using compressed fluids. Flexible substrates, not planar. OLED. Fluid flow regulation. Piston. figures 1a,1d,1e figures 4a,4b,5 paragraphs '0003!, '0008! paragraphs '0010! - '0014! paragraphs '0028!, '0035!	1-29

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US2005/014888

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