(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) **EP 1 212 386 B1**

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention of the grant of the patent:
10.08.2016 Bulletin 2016/32

(21) Application number: **00956699.3**

(22) Date of filing: **30.08.2000**

(51) Int Cl.:
C09K 11/06 *(2006.01)* ***H05B 33/14*** *(2006.01)*

(86) International application number:
PCT/GB2000/003349

(87) International publication number:
WO 2001/016251 (08.03.2001 Gazette 2001/10)

(54) METHOD FOR DEPOSITING A LIGHT-EMITTING POLYMER LAYER

METHODE ZUR ABLAGERUNG EINER LICHTEMITTIERENDEN POLYMERSCHICHT

MEHODE CONCUE POUR DEPOSER UNE COUCHE POLYMERE ELECTROLUMINESCENTE

(84) Designated Contracting States:
AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL

(30) Priority: **31.08.1999 GB 9920543**

(43) Date of publication of application:
12.06.2002 Bulletin 2002/24

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EP 1 212 386 B1

Printed by Jouve, 75001 PARIS (FR)

Description

[0001] The present invention relates to a method of depositing a conjugated polymer layer, particularly a conjugated polymer layer for a light-emitting device (LED).

[0002] Light-emitting devices using as the light-emitting layer a semiconductive conjugated polymer are known. Figure 1 shows the construction of a simple light-emitting device. A glass or plastics substrate 2 is coated with an anode layer 4, for example in the form of indium tin oxide. The anode can be patterned in the form of elongate strips. The anode layer may be coated with a hole transport layer. A light-emitting polymer layer 6 is then deposited followed by the deposition of an electron transport layer. The device structure is then completed by the deposition of a cathode layer 8. By way of example, the cathode layer can be calcium or aluminium. The cathode layer 8 can be patterned in crosswise strips to define pixels P where the anode and cathode overlap. Alternatively with an unpatterned cathode, light emitting strips can be defined. Further alternatively, the pixels may be defined on an active matrix back-plane and the cathode may be patterned or unpatterned. When an electric field is applied between the anode and cathode, holes and electrons are injected into the light-emitting polymer layer 6. The holes and electrons recombine in the polymer layer and a proportion decay radiatively to generate light.

[0003] The hole transport layer can be comprised generally of any compound capable of sustaining hole transport. Examples of suitable materials are organic conductors such as the following conducting polymers: polyaniline, polyethylenedioxythiophene and other polythiophenes, polypyrrole, etc. in their doped forms. Other alternative materials are conjugated polyamines and also low molecular weight amines such as TPD.

[0004] The light-emissive layer may comprise any molecular or polymeric compounds which are capable of sustaining charge carrier transport and also capable of light emission under device driving conditions. Examples include fluorescent organic compounds and conjugated polymers such as Alq3, polyphenylenes and derivatives, polyfluorenes and derivatives, polythiophenes and derivatives, polyphenylene vinylenes and derivatives, polymers containing heteroaromatic rings, etc..

[0005] The electron transport layer may generally comprise any material capable of sustaining electron transport. Examples include perylene systems, Alq3, polyfluorenes or polyfluorene copolymers, polymers containing heteroaromatic rings, etc..

[0006] The device may contain any combination of the above layers provided it includes at least one light-emissive layer.

[0007] OLEDs are described in US Patent No. 5,247,190 or in US Patent No. 4,539,507. In US 5,247,190 the active organic layer is a light-emissive semiconductive conjugated polymer and in US 4,539,507 the active organic layer is a light-emissive sublimed molecular film.

[0008] Conventionally, the polymer layer is deposited by spin-coating or metered blade-coating a polymer solution onto the anode and then either allowing the solvent to evaporate at RTP, or driving off the solvent using heat treatment and/or reduced pressure. The polymer can be the light-emitting polymer itself cast directly from solution, or a precursor to the polymer, which is converted to the light-emitting polymer during a heat treatment step. The polymer layer can comprise a blend of two or more materials, such as a blend of two or more polymers.

[0009] WO99/13692, US5912473 and US5777070 each describe the use of xylene to deposit a film of conjugated polymer by spin-coating. EP0923083A1 describes the use of a solvent including xylene for forming a film of DNSA-doped polyaniline, and also describes the use of gum terpentine to remove such a film from its substrate. EP0825242A2 refers to xylene as an example of a good solvent for a polymeric fluorescent substance.

[0010] The present applicants realised that it is possible to utilise a different deposition technique for depositing the polymer layer in a light-emitting device, as outlined in GB2336553. According to GB2336553, the light-emitting polymer is deposited by a technique the same as or similar to inkjet printing by supplying a solution-processible material including the polymer through a plurality of elongate bores, either through the effect of gravity or under pressure or utilising the effect of surface tension. This facilitates direct deposition or patterning of the polymer films as required. Figure 2 illustrates the essence of the technique.

[0011] It is important to use material formulations with which thin polymer films exhibiting excellent emission uniformity can be produced. In this respect, it is important to use formulations which exhibit the desired properties with respect to surface tension, viscosity, concentration, and contact angle (on the depositing medium and the substrate on to which it is to be deposited), and which preferably also exhibit good solution stability.

[0012] It is an aim of the present invention to provide formulations which facilitate the supply thereof onto a substrate via a plurality of elongate bores to form patterned polymer films. In particular, it is an aim of the present invention to provide a formulation with which polymer films which exhibit improved emission uniformity can be deposited, particularly in the context of relatively high molecular weight polymers with intrinsically rigid conjugated systems.

[0013] According to the present invention, there is provided a method according to claim 1.

[0014] Preferred embodiments of the invention are defined in claims 2 to 23.

[0015] The solvent used in the formulation preferably consists substantially of at least one substance selected from the group of substances recited in claim 1. In a preferred embodiment, it consists substantially of a blend of two or more solvents belonging to this group.

[0016] Monoterpenes are particularly preferred such as terpinolene, limonene and α-terpineol.

[0017] The terpene is in liquid form at the deposition temperature.

[0018] Preferred polyalkylated aromatic compounds include polyalkyl benzenes such as cymene and isodurene. It is preferred that each of the alkyl substituents on the aromatic ring are distinct from each other, i.e. that they are only bonded together via the aromatic ring.

[0019] According to one embodiment, the solvent comprises at least one aromatic compound substituted with alkyl groups at no less than 3 positions.

[0020] For the purposes of this application, the term conjugated polymer refers to polymers, including oligomers such as dimers, trimers etc., which are fully conjugated (i.e. are conjugated along the entire length of the polymer chain) or are partially conjugated (i.e. which include non-conjugated segments in addition to conjugated segments).

[0021] The polymer may be a polymer suitable for use in a light-emissive layer, a hole transport layer or an electron transport layer in an organic light-emitting device.

[0022] In a preferred example, the conjugated polymer can be a light-emitting polymer, hole transport polymer or electron transport polymer itself, or a precursor to a light-emitting polymer, hole transport polymer or electron transport polymer. The conjugated polymer or its precursor can be any suitable polymer, and in particular can be any one of the following:

a) conducting polymers such as polyaniline (PANI) and derivatives, polythiophenes and derivatives, polypyrrole and derivatives, polyethylene dioxythiophene; doped forms of all these and particularly polystyrene sulphonic acid-doped polyethylene dioxythiophene (PEDT/PSS);

b) solution processible charge transporting and/or luminescent/electro-luminescent polymers, preferably conjugated polymers such as: polyphenylenes and derivatives, polyphenylene vinylenes and derivatives, polyfluorenes and derivatives, tri-aryl containing polymers and derivatives, precursor polymers in various forms, copolymers (including the above-named polymer classes), generally random and block copolymers, polymers with the active (charge transporting and/or luminescent) species attached as side-groups to the main chain, thiophenes and derivatives, etc..

[0023] The specific example discussed herein is a blend of 5BTF8 (80% in weight) and TFB (20%).

[0024] For a better understanding of the invention and to show how the same may be carried into effect, reference will now be made by way of example to the accompanying drawings in which:

Figure 1 is a diagram of a light-emitting device;
Figure 2 is a diagram illustrating a method of depositing various polymer layers;
Figures 3a to 3e represent the structures of isodurene, terpinolene, limonene cymene and a-terpineol respectively;
Figures 4a to 4c illustrate the formulae of TFB and 5BTF8;
Figure 5 is a graph of optical density versus time illustrating the improved solubility stability of the solvents discussed herein;
Figure 6 is a graph showing the photoluminescent properties of a polymer layer deposited in xylene and those of one deposited in isodurene; and
Figure 7 is a graph showing the photoluminescent properties of polymer layers deposited in isodurene-terpinolene (3:1) and xylene.

[0025] Figure 2 illustrates a deposition technique for depositing a polymer layer 6 onto the patterned anode 4. A plurality of elongate bores 10 are illustrated, each aligned with a respective trough 8. The elongate bores 10 are connected via a conduit 12 to a reservoir 14 holding the solution to be deposited. The solution is supplied through the elongate bores 10 under pressure or by gravity to deposit the light emitting polymer layer 6. Specific formulations for the solution processible material 16 for use in a method according to the claimed invention are discussed herein.

Example 1

[0026] The first exemplified formulation is 0.5% W/V of 5BTF8 (80% in weight) and TFB (20%) in isodurene. The structure of TFB is illustrated in Figure 4a. 5BTF8 is F8 (structure of Figure 4b) doped with 5% F8BT (structure of Figure 4c).

Example 2

[0027] According to the second example, the formulation comprises 0.5% W/V of 5BTF8 (80% in weight) and TFB (20%) in a solvent comprising a blend of isodurene:terpinolene (3:1).

Example 3

[0028] According to a third example, the formulation comprises 0.5% W/V of 5BTF8 (80% in weight) and TFB (20%) in isodurene:limonene (3:1).

Example 4

[0029] According to a fourth example, the formulation comprises 0.5% W/V of 5BTF8 (80% in weight) and TFB (20%) in isodurene:cymene (3:1).

[0030] Using the formulation according to each example, a polymer layer was deposited using the technique described in relation to Figure 2. The solubilities were measured with over a 48 hour period at 17°C. The results are plotted in Figure 5, from which it can readily be seen

that they display excellent stability of solubility over that period.

[0031] Subsequently, the photoluminescent properties of Examples 1 and 2 were compared with photoluminescent properties of a polymer deposited using a xylene solvent (which is another example of a solvent for use in a method according to the claimed invention). Figure 6 illustrates the results of Example 1, where the solvent is isodurene and Figure 7 illustrates the results for Example 2 where the solvent is isodurene-terpinolene (3:1). Figure 6 and 7 also illustrate the case where the polymer layer is heat-treated after deposition.

Claims

1. A method of depositing a polymer layer in a light emitting device by supplying a solution processible formulation via a plurality of elongate bores (10) onto a substrate including a layer of a doped polymer as an organic conductor, wherein the formulation comprises a conjugated polymer dissolved in a solvent, the solvent comprising at least one substance selected from the group consisting of terpenes or terpenes substituted with one or more groups selected from alcohol, ester, ether, ketone and aldehyde groups and polyalkylated aromatic compounds.

2. A method according to claim 1 wherein the terpene is a monoterpene.

3. A method according to claim 1 or claim 2, wherein the solvent comprises a terpene substituted with one or more groups selected from alcohol, ester, ether, ketone and aldehyde groups.

4. A method according to claim 2 or claim 3 wherein the solvent comprises a substance selected from the group consisting of terpinolene, limonene and α-terpineol.

5. A method according to claim 1 wherein the polyalkylated aromatic compound is a polyalkylated benzene.

6. A method according to claim 5 wherein the polyalkylated benzene is a dialkylbenzene.

7. A method according to claim 6 wherein the dialkylbenzene is selected from the group consisting of cymene and xylene.

8. A method according to claim 5 wherein the polyalkylated benzene is a tetraalkylbenzene.

9. A method according to claim 8 wherein the tetraalkylbenzene is isodurene.

10. A method according to any preceding claim, wherein the polymer is a light-emissive polymer.

11. A method according to claim 1 wherein the polymer comprises a fluorene-based polymer.

12. A method according to claim 1 wherein the polymer comprises a polymer containing fluorene and triarylamine units.

13. A method according to claim 1 wherein the polymer comprises a blend of a fluorene-based polymer and a polymer containing fluorene and triarylamine units.

14. A method according to claim 13 wherein the polymer is a blend of 5BTF8 and TFB.

15. A method according to claim 14, wherein the constituents of the polymer blend are 5BTF8 (80%); TFB (20%).

16. A method according to any preceding claim, wherein the solvent comprises a blend of two or more of said substances.

17. A method according to claim 16 wherein the blend comprises two or more substances selected from the solvent group consisting of isodurene, cymene, terpinolene, limonene and a-terpineol.

18. A method according to claim 17, wherein the blend contains 3 parts isodurene to 1 part of another member of the solvent group.

19. A method according to claim 18, wherein the blend is isodurene:terpinolene (3:1).

20. A method according to claim 18, wherein the blend is isodurene:limonene (3:1).

21. A method according to claim 18, wherein the blend is isodurene:cymene (3:1).

22. A method according to any preceding claim wherein the solution processible formulation is supplied by ink-jet printing.

23. A method according to any preceding claim wherein the substrate includes a layer of doped polyethylenedioxythiophene.

Patentansprüche

1. Verfahren zum Abscheiden einer Polymerschicht in einer lichtemittierenden Vorrichtung durch Zuführen einer in Lösung verarbeitbaren Formulierung mittels mehrerer länglicher Bohrlöcher (10) auf ein Substrat,

das eine Schicht eines dotierten Polymers als einen organischen Leiter beinhaltet, wobei die Formulierung ein konjugiertes Polymer umfasst, das in einem Lösungsmittel gelöst ist, wobei das Lösungsmittel mindestens eine Substanz umfasst, die aus der Gruppe bestehend aus Terpenen oder Terpenen, die mit einer oder mehreren Gruppen substituiert sind, die aus Alkohol-, Ester-, Ether-, Keton- und Aldehydgruppen ausgewählt sind, und polyalkylierten aromatischen Verbindungen ausgewählt ist.

2. Verfahren nach Anspruch 1, wobei das Terpen ein Monoterpen ist.

3. Verfahren nach Anspruch 1 oder 2, wobei das Lösungsmittel ein Terpen umfasst, das mit einer oder mehreren Gruppen substituiert ist, die aus Alkohol-, Ester-, Ether-, Keton- und Aldehydgruppen ausgewählt sind.

4. Verfahren nach Anspruch 2 oder 3, wobei das Lösungsmittel eine Substanz umfasst, die aus der Gruppe bestehend aus Terpinolen, Limonen und α-Terpineol ausgewählt ist.

5. Verfahren nach Anspruch 1, wobei die polyalkylierte aromatische Verbindung ein polyalkyliertes Benzol ist.

6. Verfahren nach Anspruch 5, wobei das polyalkylierte Benzol ein Dialkylbenzol ist.

7. Verfahren nach Anspruch 6, wobei das Dialkylbenzol aus der Gruppe bestehend aus Cymol und Xylol ausgewählt ist.

8. Verfahren nach Anspruch 5, wobei das polyalkylierte Benzol ein Tetraalkylbenzol ist.

9. Verfahren nach Anspruch 8, wobei das Tetraalkylbenzol Isoduren ist.

10. Verfahren nach einem vorhergehenden Anspruch, wobei das Polymer ein lichtemittierendes Polymer ist.

11. Verfahren nach Anspruch 1, wobei das Polymer ein fluorenbasiertes Polymer umfasst.

12. Verfahren nach Anspruch 1, wobei das Polymer ein Polymer umfasst, das Fluoren- und Triarylamin-Einheiten enthält.

13. Verfahren nach Anspruch 1, wobei das Polymer ein Gemisch eines fluorenbasierten Polymers und eines Polymers, das Fluoren- und Triarylamin-Einheiten enthält, umfasst.

14. Verfahren nach Anspruch 13, wobei das Polymer ein Gemisch von 5BTF8 und TFB ist.

15. Verfahren nach Anspruch 14, wobei die Bestandteile des Polymergemischs 5BTF8 (80 %); TFB (20 %) sind.

16. Verfahren nach einem vorhergehenden Anspruch, wobei das Lösungsmittel ein Gemisch von zwei oder mehr der Substanzen umfasst.

17. Verfahren nach Anspruch 16, wobei das Gemisch zwei oder mehr Substanzen umfasst, die aus der Lösungsmittelgruppe bestehend aus Isoduren, Cymol, Terpinolen, Limonen und α-Terpineol ausgewählt sind.

18. Verfahren nach Anspruch 17, wobei das Gemisch 3 Teile Isoduren zu 1 Teil eines anderen Mitglieds der Lösungsmittelgruppe enthält.

19. Verfahren nach Anspruch 18, wobei das Gemisch Isoduren:Terpinolen (3:1) ist.

20. Verfahren nach Anspruch 18, wobei das Gemisch Isoduren:Limonen (3:1) ist.

21. Verfahren nach Anspruch 18, wobei das Gemisch Isoduren:Cymol (3:1) ist.

22. Verfahren nach einem vorhergehenden Anspruch, wobei die in Lösung verarbeitbare Formulierung durch Tintenstrahldruck zugeführt wird.

23. Verfahren nach einem vorhergehenden Anspruch, wobei das Substrat eine Schicht von dotiertem Polyethylendioxythiophen beinhaltet.

Revendications

1. Un procédé consistant à déposer une couche de polymère dans un dispositif électroluminescent en fournissant une préparation pouvant être traitée en solution via une pluralité d'alésages allongés (10) sur un substrat incluant une couche de polymère dopé en tant que conducteur organique, dans lequel la préparation comprend un polymère conjugué dissous dans un solvant, le solvant comprenant une ou plusieurs substances sélectionnées dans le groupe constitué par des terpènes ou des terpènes substitués avec un ou plusieurs groupes sélectionnés dans les groupes d'alcools, d'esters, d'éthers, de cétones et d'aldéhydes et des composés aromatiques polyalkylés.

2. Un procédé selon la revendication 1 dans lequel le terpène est un monoterpène.

3. Un procédé selon la revendication 1 ou la revendication 2, dans lequel le solvant comprend un terpène substitué avec un ou plusieurs groupes sélectionnés dans les groupes d'alcools, d'esters, d'éthers, de cétones et d'aldéhydes.

4. Un procédé selon la revendication 2 ou la revendication 3 dans lequel le solvant comprend une substance sélectionnée dans le groupe constitué par le terpinolène, le limonène et le α-terpinéol.

5. Un procédé selon la revendication 1 dans lequel le composé aromatique polyalkylé est un benzène polyalkylé.

6. Un procédé selon la revendication 5 dans lequel le benzène polyalkylé est un dialkylbenzène.

7. Un procédé selon la revendication 6 dans lequel le dialkylbenzène est sélectionné dans le groupe constitué par le cymène et le xylène.

8. Un procédé selon la revendication 5 dans lequel le benzène polyalkylé est un tétraalkylbenzène.

9. Un procédé selon la revendication 8 dans lequel le tétraalkylbenzène est de l'isodurène.

10. Un procédé selon l'une quelconque des revendications précédentes, dans lequel le polymère est un polymère électroluminescent.

11. Un procédé selon la revendication 1 dans lequel le polymère comprend un polymère à base de fluorène.

12. Un procédé selon la revendication 1 dans lequel le polymère comprend un polymère contenant des unités de fluorène et de triarylamine.

13. Un procédé selon la revendication 1 dans lequel le polymère comprend un mélange d'un polymère à base de fluorène et d'un polymère contenant des unités de fluorène et de triarylamine.

14. Un procédé selon la revendication 13, dans lequel le polymère est un mélange de 5BTF8 et de TFB.

15. Un procédé selon la revendication 14, dans lequel les constituants du mélange de polymère sont le 5BTF8 (80 %) et le TFB (20 %).

16. Un procédé selon l'une quelconque des revendications précédentes, dans lequel le solvant comprend un mélange de deux ou plus de deux desdites substances.

17. Un procédé selon la revendication 16 dans lequel le mélange comprend deux ou plus de deux substances sélectionnées dans le groupe de solvants constitué par l'isodurène, le cymène, le terpinolène, le limonène et le α-terpinéol.

18. Un procédé selon la revendication 17, dans lequel le mélange contient 3 parties d'isodurène pour 1 partie d'un autre élément du groupe de solvants.

19. Un procédé selon la revendication 18, dans lequel le mélange est de l'isodurène et du terpinolène (3:1).

20. Un procédé selon la revendication 18, dans lequel le mélange est de l'isodurène et du limonène (3:1).

21. Un procédé selon la revendication 18, dans lequel le mélange est de l'isodurène et du cymène (3:1).

22. Un procédé selon l'une quelconque des revendications précédentes, dans lequel la préparation pouvant être traitée en solution est fournie par impression par jet d'encre.

23. Un procédé selon l'une quelconque des revendications précédentes, dans lequel le substrat inclut une couche de polyéthylènedioxythiophène dopé.

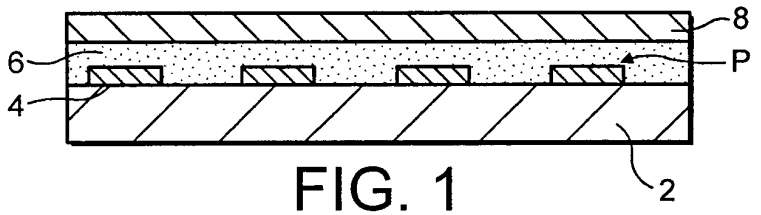


FIG. 1

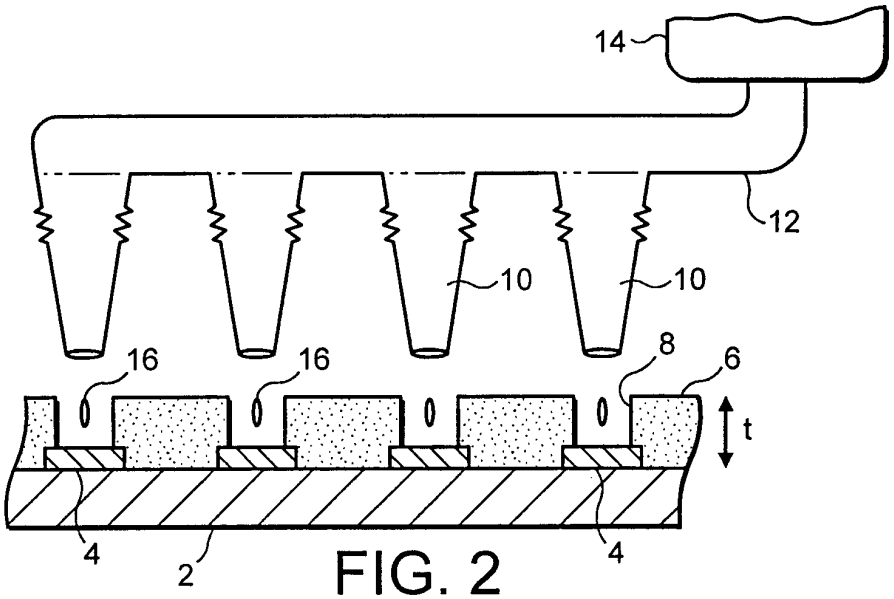
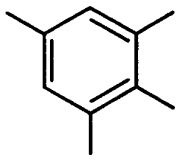
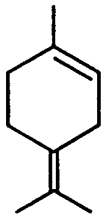


FIG. 2



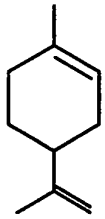
ISODURENE

FIG. 3a



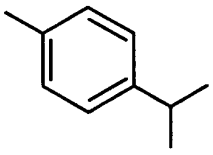
TERPINOLENE

FIG. 3b



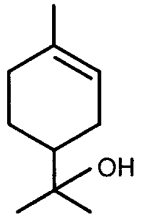
LIMONENE

FIG. 3c



CYMENE

FIG. 3d



α-TERPINEOL

FIG. 3e

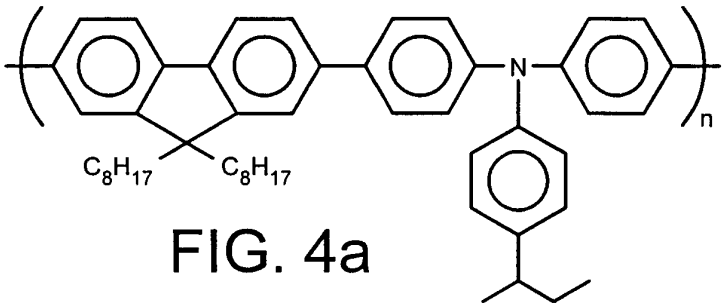


FIG. 4a

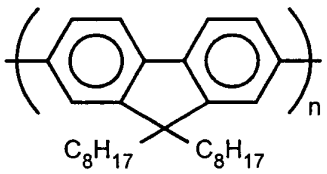


FIG. 4b

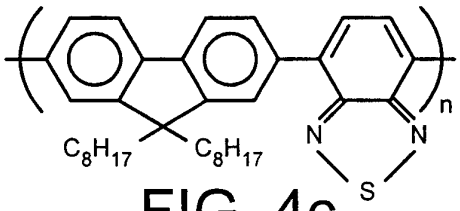


FIG. 4c

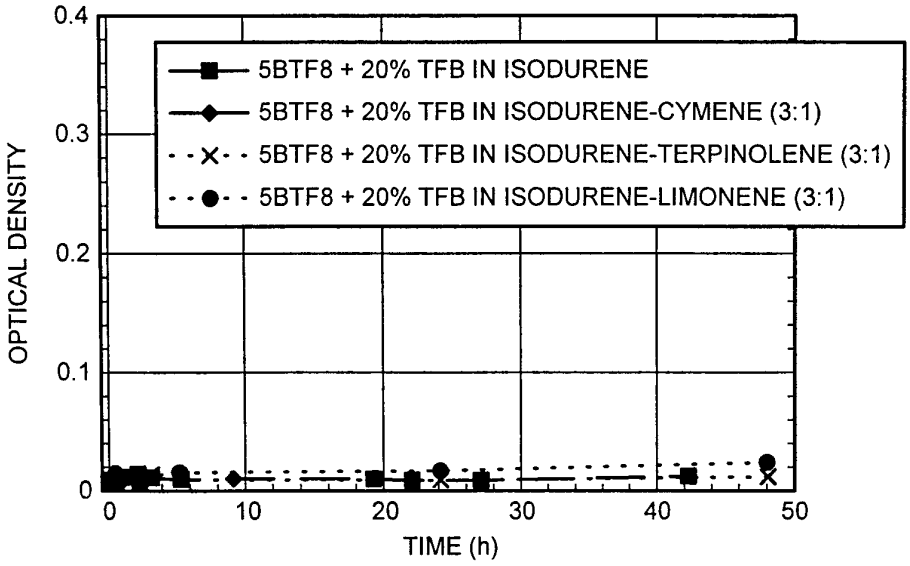


FIG. 5

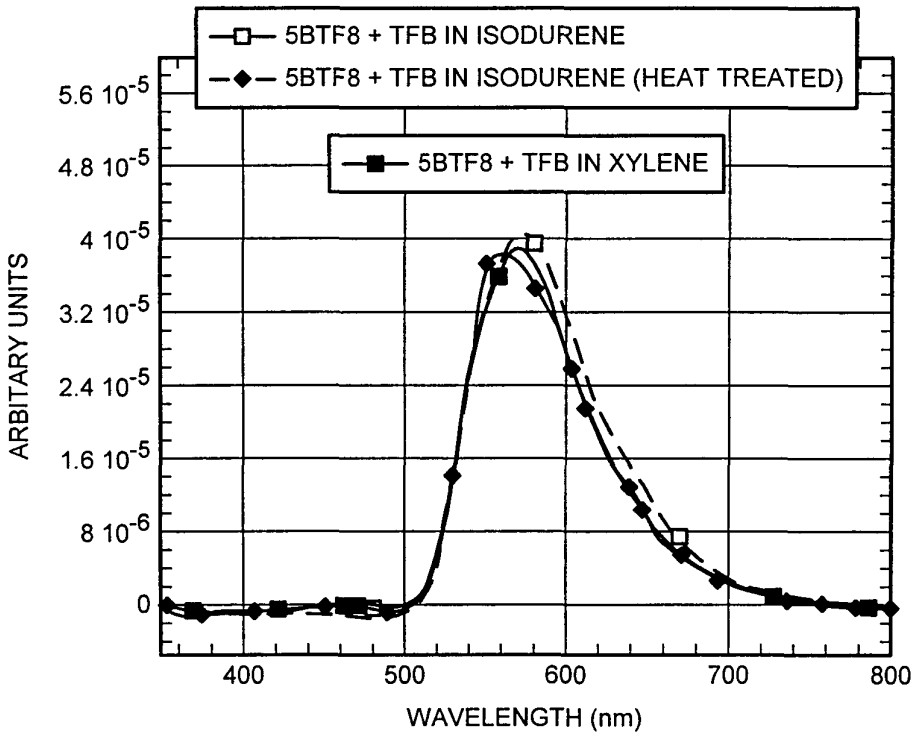

5BTF8 + TFB IN ISODURENE
5BTF8 + TFB IN ISODURENE (HEAT TREATED)
5BTF8 + TFB IN XYLENE
ARBITARY UNITS
5.6 10-5
4.8 10-5
4 10-5
3.2 10-5
2.4 10-5
1.6 10-5
8 10-6
0
400
500
600
700
800
WAVELENGTH (nm)

FIG. 6

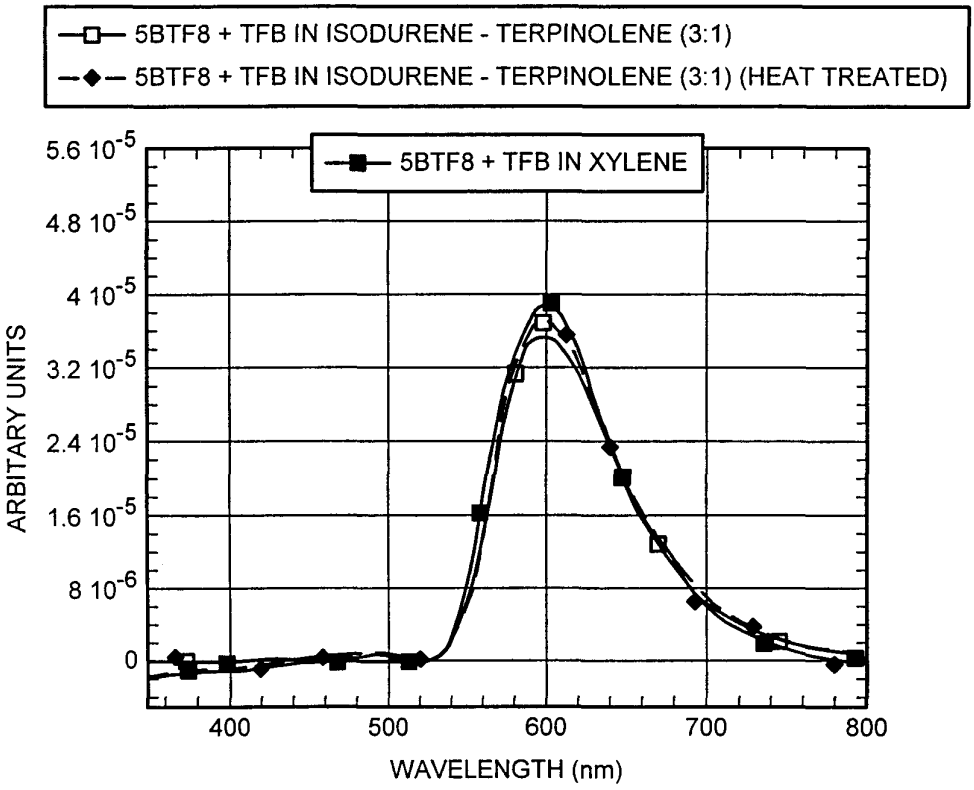

5BTF8 + TFB IN ISODURENE - TERPINOLENE (3:1)
5BTF8 + TFB IN ISODURENE - TERPINOLENE (3:1) (HEAT TREATED)
5BTF8 + TFB IN XYLENE
ARBITARY UNITS
5.6 10-5
4.8 10-5
4 10-5
3.2 10-5
2.4 10-5
1.6 10-5
8 10-6
0
400
500
600
700
800
WAVELENGTH (nm)

FIG. 7

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

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- US 4539507 A **[0007]**
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- EP 0825242 A2 **[0009]**
- GB 2336553 A **[0010]**

专利名称(译)	沉积发光聚合物层的方法		
公开(公告)号	EP1212386B1	公开(公告)日	2016-08-10
申请号	EP2000956699	申请日	2000-08-30
[标]申请(专利权)人(译)	剑桥显示技术有限公司 精工爱普生株式会社		
申请(专利权)人(译)	剑桥显示科技有限公司 SEIKO EPSON CORPORATION		
当前申请(专利权)人(译)	剑桥显示科技有限公司 SEIKO EPSON CORPORATION		
[标]发明人	GRIZZI ILARIA CAMBRIDGE DISPLAY TECH LTD		
发明人	GRIZZI, ILARIA, CAMBRIDGE DISPLAY TECHNOLOGY LTD		
IPC分类号	C09K11/06 H05B33/14 C09J201/00 H01L51/00 H01L51/30 H01L51/40 H01L51/50 H05B33/10		
CPC分类号	C09K11/06 C09K2211/1416 C09K2211/1425 C09K2211/1433 C09K2211/1483 H01L51/0005 H01L51/0007 H01L51/0036 H01L51/0039 H01L51/0043 H01L51/0059 H01L51/5012 H05B33/10 H05B33/14		
优先权	1999020543 1999-08-31 GB		
其他公开文献	EP1212386A1		
外部链接	Espacenet		

摘要(译)

一种用于在发光器件中沉积聚合物层的配方，该配方包含溶解在溶剂中的共轭聚合物，该溶剂包含至少一种选自萜烯和多烷基化芳族化合物的物质。

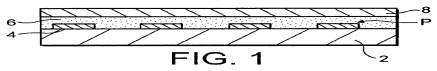

FIG. 1

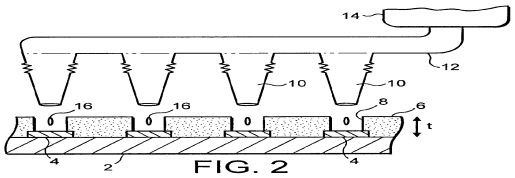

FIG. 2

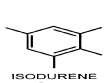

FIG. 3a

FIG. 3b

FIG. 3c

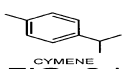

FIG. 3d

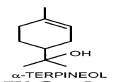

FIG. 3e