结构简单的高效蓝色荧光器件

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摘要 热激活延迟荧光(thermally activated delayed fluorescence, TADF)材料双[4-(9,9-二甲基-9,10-二氢吡啶)苯基]硫磺(DMAC-DPS)在非掺杂型器件中展示了良好的发光特性. 本文首先以DMAC-DPS作为器件的发光层, 分别对其厚度以及器件的空穴传输层和电子传输层进行了优化, 制备了结构简单的非掺杂型高效蓝光器件, 其最大外量子效率是14.3%, 最大功率效率是 26.8 lm W^{-1} . 荧光器件在稳定性等方面具备显著的优越性且荧光材料已被广泛采用, 因此我们进一步以DMAC-DPS为主体材料, 蓝色荧光材料2,5,8,11-四叔丁基芘(TBPe)为客体材料制备了高效的TADF材料敏化蓝光器件. 器件的最大外量子效率为12.7%, 最大功率效率为 22.9 lm W^{-1} . 我们的结果表明分别以TADF材料作为发光材料和主体材料可以制备高效蓝色荧光器件, 这为结构简单高效蓝光器件的制备提供了一种新的途径.

关键词 热激活延迟荧光, 有机蓝光二极管, 敏化, 主体材料

有机发光二极管(organic light-emitting diodes, OLEDs)在电激发时产生比例为1:3的单线态激子和三线态激子. 基于传统荧光材料的器件中三线态激子由于自旋禁阻不能发光而限制了器件的内量子效率. 因此, 如何充分利用三线态激子发光成为人们研究的热点.

磷光材料中一般包含铱、铂等重金属元素, 它们的引入提高了自旋-轨道耦合作用, 使三线态激子有了单线态激子的特性, 导致三线态激子能向基态辐射跃迁从而使基于磷光材料的器件内量子效率可达100%. 铱、铂等重金属的引入使磷光材料价格较高, 特别是蓝色磷光器件驱动稳定性较差无法实际应用等亟待解决的问题迫使人们寻找它们的有效替代品. TADF材料因其在分子结构不包含铱、铂等重金属元素的情况下能够利用三线态激子发光而成为继传统荧光材料、磷光材料之后的第三代有机发光材

料. 这是因为TADF材料拥有较小的单线态-三线态能级差(ΔE_{ST}), 使得三线态激子能有效地通过反向系间窜越(reverse intersystem crossing, RISC)形成单线态激子继而发光. 自2012年Adachi研究组^[1]首次报道了高效率TADF材料以来, 人们对TADF发光材料和器件的研究取得了长足的进步. TADF绿光器件的外量子效率已经接近30%^[2]. 近来, 基于TADF材料敏化荧光的有机发光器件成为新的研究方向. Nakanotani等人^[3]以TADF材料作为辅助掺杂剂制备了外量子效率为13.4%~18%的蓝色、绿色、黄色和红色荧光发光器件, 采用这种方法可以利用普遍使用的有机荧光材料来制备高效率的发光器件.

2014年, Adachi研究组^[4]报道了高效蓝色TADF材料DMAC-DPS, 基于DMAC-DPS的器件外量子效率高达19.5%. DMAC-DPS发光器件多采用主客体掺杂的方式制备, 器件结构复杂. 已报道的以DMAC-

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DPS为发光材料的器件采用多种组合形式的空穴传输层(hole transport layer, HTL): 既有不含HTL或HTL仅是1,3-二-9-咔唑基苯(mCP)^[5], 还有HTL为双材料N,N'-二(α-萘基)-N,N'-二苯基-4,4'-联苯胺(α-NPD)和mCP的组合^[6], 以及三种材料4,4'-环己基二[N,N'-二(4-甲基苯基)苯胺](TAPC)、4,4',4''-三(咔唑-9-基)三苯胺(TCTA)和mCP的组合^[7]. 采用多种材料组合的HTL无疑会增加器件制备的复杂性, 不同空穴传输层对器件特性的影响还有待进一步研究. 具有简单结构的非掺杂型高效率发光器件能够降低器件成本和提高器件的产率, 还鲜有报道. Zhang等人^[5]研究发现DMAC-DPS具有较大的斯托克斯位移和较弱的分子间相互作用导致其光致发光量子产率(photoluminescence quantum yields, PLQY)对掺杂浓度并不敏感, DMAC-DPS薄膜的PLQY高达0.88. 他们利用DMAC-DPS制备了外量子效率高达19.5%的非掺杂型蓝光器件, 但器件的驱动电压较高, 在亮度为1000 cd m⁻²时电压约为10 V. 高效率的DMAC-DPS蓝光器件引起广泛关注, Song等人^[7]以DMAC-DPS及荧光材料2,8-二叔丁基-5,11-二(4-叔丁基苯基)-6,12-苯基四并苯(TBRb)双客体共掺杂的方法制备了外量子效率为17.6%, 功率效率为41.0 lm W⁻¹的白光器件, 进而他们又采用DMAC-DPS和磷光材料双(4-苯并噻吩[3,2-C]吡啶-N,C^{2'})乙酰丙酮合铱(PO-01)制备了外量子效率为22.4%, 功率效率高达60.3 lm W⁻¹的白光器件^[8], 但这些研究都是采用DMAC-DPS作为蓝色发光材料同黄光材料匹配来获得白光器件.

本文研究了基于DMAC-DPS结构简单的非掺杂型高效蓝光器件, 分别对器件各功能层进行了优化,

得到了外量子效率为14.3%, 功率效率为26.8 lm W⁻¹的非掺杂型蓝光器件. 在此基础上以DMAC-DPS为主体材料, 蓝色荧光材料TBPe为客体材料制备了蓝光器件, 蓝光器件外量子效率和功率效率分别为12.7%和22.9 lm W⁻¹.

1 器件制备与测量

图1给出了所用有机材料的化学式及器件的能级结构. 其中N,N'-二苯基-N,N'-(1-萘基)-1,1'-联苯-4,4'-二胺(NPB)、mCP是空穴传输材料, 二[2-((氧代)二苯基磷基)苯基]醚(DPEPO)、2,7-双(二苯基磷)-9,9'-螺二[芴](SPPO13)、1,3,5-三[(3-吡啶基)-3-苯基]苯(TmPyPB)为电子传输材料, DMAC-DPS和TBPe为发光材料, 三氧化钼(MoO₃)为空穴注入材料, 氟化铯(CsF)为电子注入材料. 各有机材料的最高占据态分子轨道(HOMO)和最低未占据分子轨道(LUMO)能级值引自文献[3,7,9~11]. 实验所用材料和铟锡氧化物(ITO)导电玻璃衬底通过商业途径购买. 在使用导电玻璃衬底之前要对其进行彻底的清洗. 首先使用清洗液(Decon 90, 浓度5%)对ITO衬底进行多次擦拭, 然后依次使用去离子水、无水乙醇和丙酮分别水浴超声(水温约60℃)两次, 然后烘干备用.

器件中所用的有机及无机材料层均在真空度低于1.0×10⁻⁴ Pa的真空环境下蒸镀制备, 各功能层的厚度及沉积速率由膜厚测试仪精确控制. 器件的电压-电流密度-亮度(V-I-B)特性采用Labview程序控制的Keithley 2400数字精密电源和色度计Chroma Meter CS-100A进行测量. 发光光谱由Ocean Optics USB4000-UV-VIS光纤光谱仪测得.

图1 材料的分子结构式及器件能级结构图

Figure 1 Chemical structures of the materials and the energy level diagram of devices

2 结果分析与讨论

2.1 不同空穴传输层对器件性能的影响

首先对不同HTL对器件性能的影响进行了研究. 制备了结构分别为(A): ITO/MoO₃(5 nm)/NPB (30 nm)/mCP(10 nm)/DMAC-DPS(30 nm)/DPEPO(10 nm)/TmPyPB(40 nm)/CsF(1 nm)/Al和(B): ITO/MoO₃ (5 nm)/mCP(40 nm)/DMAC-DPS(30 nm)/DPEPO(10 nm)/TmPyPB(40 nm)/CsF(1 nm)/Al的器件. 从图2中可以看出器件(A)的电流密度及亮度都低于器件(B),

且其启亮电压比后者稍高. MoO₃的功函数为6.6 eV, 能够有效地将空穴注入到NPB和mCP. 虽然NPB具有大于mCP的空穴迁移率^[12,13], 但NPB与mCP的HOMO之间存在约0.7 eV的能级差, 不利于空穴从NPB到mCP的传输^[14], 影响器件的电流密度. 但器件中空穴为多数载流子, 因此空穴注入和传输情况的改变对器件发光效率影响较小, 两类器件具有接近的发光效率. 表1给出了器件的特性. 两者的电致发光(electroluminescence, EL)光谱中发光峰位均位于480 nm处, 为DMAC-DPS的发光, 发光的色坐标为(0.16, 0.29).

图2 (网络版彩色)NPB/mCP或mCP作为HTL器件的V-I-B特性(a), 电流密度-效率特性(b)及EL光谱图(c)

Figure 2 (Color online) Characteristics of the devices using NPB/mCP or mCP as HTL: V-I-B plots (a), current density-efficiency curves (b) and electroluminescence spectra (c)

表1 器件的特性

Table 1 Device performances of devices

器件	最大效率			亮度为 1000 cd m ⁻² 时的效率			色坐标
	CE (cd A ⁻¹)	PE (lm W ⁻¹)	EQE (%)	CE (cd A ⁻¹)	PE (lm W ⁻¹)	EQE (%)	
(A)	29.8	21.3	16.1	16.9	6.8	9.1	(0.16, 0.29)
(B)	30.0	26.2	16.2	19.0	8.5	10.3	(0.16, 0.28)
(C)	25.2	22.2	13.8	12.5	6.4	6.8	(0.16, 0.29)
(D)	25.6	26.8	14.3	14.9	10.4	8.3	(0.16, 0.27)
(E)	21.3	22.3	12.5	10.9	5.7	6.4	(0.15, 0.25)
(F)	21.9	22.9	12.7	12.8	8.5	7.4	(0.15, 0.25)
(G)	20.2	21.1	11.3	13.4	8.3	7.5	(0.15, 0.26)

2.2 不同电子传输层对器件性能的影响

制备了结构为ITO/MoO₃(5 nm)/mCP(40 nm)/DMAC-DPS(30 nm)/ETL/CsF(1 nm)/Al的发光器件, 其中电子传输层(electron transport layer, ETL)分别为DPEPO(10 nm)/TmPyPB(40 nm), TmPyPB(50 nm)和SPPO13(50 nm), 对应器件分别为(B), (C)和(D)来研究不同ETL对器件性能的影响. 图3给出了器件的*V-I-B*(a), 电流密度-效率(b)及EL光谱(c)特性. 从中可看出, 相同电压下器件(D)的电流密度最大, 器件(B)的电流密度最小. DPEPO的HOMO和LUMO能级(见图1), 对器件中载流子的传输产生一定的阻挡作用. 另外, 其本身较低电子迁移率^[15]一定程度上限制了电子的传输. 文献中报道的以50 nm DPEPO为ETL的器件启亮电压为4.3 V, 在17 V时电流密度仅为50 mA cm⁻²^[5]. 器件(B)采用10 nm DPEPO作为激子阻挡层, 其启亮电压和驱动电压明显降低. 相比之下, 器件(C)和(D)的驱动电压进一步降低, 尤其是器件(D)的启亮电压(亮度为1 cd m⁻²)为2.7 V, 亮度达到1000 cd m⁻²时电压为4.5 V. TmPyPB和SPPO13的电子迁移率分别为1.0×10⁻³ cm² V⁻¹s⁻¹^[10]和6.4×10⁻⁵ cm² V⁻¹s⁻¹^[16], 而在相同的驱动电压下器件(D)的电流

密度更大, 这可能是由于SPPO13的能隙相较于TmPyPB更小, 高度极化的磷氧基团大大提高了阴极的电子注入效率^[11]. 另一方面, DPEPO的三线态能量为3.3 eV^[4], 能有效将形成的三线态激子限制在发光层(emitting layer, EML)内. 因此, 器件(B)的外量子效率最高, 达到16.2%. 器件(C)和器件(D)的最大外量子效率分别为13.8%和14.3%. 器件(D)较高的发光效率可能来源于器件较好的电子注入效率, 发光效率随电流密度增大而下降, 外量子效率下降到效率最大值一半时的电流密度(*J*₀)为11.3 mA cm⁻², 器件(B)和(C)的*J*₀分别为13.1及8.0 mA cm⁻². 这和文献[6]报道的以DMAC-DPS为单发光层的非掺杂型器件的*J*₀值接近. 器件(B), (C)和(D)的最高功率效率分别为26.2, 22.2和26.8 lm W⁻¹, 在亮度为1000 cd m⁻²时器件(D)功率效率为10.4 lm W⁻¹, 且在较高电流密度下的功率效率明显超过器件(B)和器件(C)的功率效率. 优化后的器件有机功能层只有3个, 极大地简化了器件结构. 采用了不同的ETL没有明显影响器件的EL谱, 器件的发光来源于DMAC-DPS.

2.3 发光层厚度对器件性能的影响

分别制备了结构为(D): ITO/MoO₃(5 nm)/mCP

图3 (网络版彩色)DPEPO/TmPyPB, TmPyPB和SPPO13 作为ETL器件的*V-I-B*特性(a), 电流密度-效率特性(b)及EL光谱图(c)

Figure 3 (Color online) Characteristics of the devices using DPEPO/TmPyPB, TmPyPB and SPPO13 as ETL: *V-I-B* plots (a), current density-efficiency curves (b) and electroluminescence spectra (c)

(40 nm)/DMAC-DPS(30 nm)/SPPO13(50 nm)/CsF (1 nm)/Al和(E): ITO/MoO₃(5 nm)/mCP(55 nm)/DMAC-DPS(15 nm)/SPPO13(50 nm)/CsF(1 nm)/Al的器件来研究了EML厚度对器件性能的影响. 器件的*V-I-B*(a)及电流密度-效率(b)特性如图4所示. 在相同电压下器件(D)具有大于器件(E)的电流密度说明DMAC-DPS的空穴传输能力超过mCP. 器件外量子效率及功率效率也都比器件(E)的略高, 这可能是由于较薄发光层对不同电压下载流子迁移率变化导致的复合区域变化等因素较敏感. 改变器件发光层的厚度并未明显改变器件的EL谱.

2.4 基于DMAC-DPS和TBPe的蓝光器件

在以上对器件优化的基础上, 制备了结构为ITO/MoO₃(5 nm)/mCP(40 nm)/DMAC-DPS: x%TBPe(30 nm)/SPPO13(50 nm)/CsF(1 nm)/Al的器件. 其中TBPe的浓度分别为1.0%(器件(F))和0.8%(器件(G)). 器件的*V-I-B*, 电流密度-效率及EL光谱特性如图5所示. 可以看到引入TBPe掺杂剂后器件的电流密度并没有明显的变化, 这和文献[17]报道的相类似, 可能是因为在较低的掺杂浓度下TBPe的空穴陷阱作用并不明显. 同样, DMAC-DPS: TBPe器件的启亮电压为2.8 V, 亮度达到1000 cd m⁻²时电压为4.8 V, 稍微高于DMAC-DPS器件的启亮电压和驱动电压. 器件(F)的外量子效率为12.7%, 功率效率为22.9 lm W⁻¹, 而器件(G)的外量子效率、功率效率分别为11.3%, 21.1 lm W⁻¹. 相比于DMAC-DPS器件, DMAC-DPS: TBPe器件的发光效率下降略为减缓, 器件(F)和(G)的*J*₀分别为12.9和19.1 mA cm⁻². DMAC-DPS: TBPe器件的EL谱发光峰位于460和489 nm处, 来源于TBPe. 器

件(F)和(G)的色坐标分别为(0.15, 0.25)和(0.15, 0.26), 器件的色度同DMAC-DPS器件相比有所改善.

以TBPe掺杂到DMAC-DPS中制备器件的外量子效率非常接近DMAC-DPS器件外量子效率(14.3%). 图6描述了DMAC-DPS以及DMAC-DPS: TBPe器件中激发态的产生和演化. 器件的激发态动力学过程可由图6中的4个过程来解释: (1) 载流子复合主要在主体材料DMAC-DPS上形成激子, 其中的单线态激子一方面直接退激发出瞬时荧光(prompt fluorescence, PF), 另一方面通过(2) Förster能量传递过程形成TBPe单线态, 其向基态跃迁得到TBPe的PF. DMAC-DPS单线态激子通过ISC过程形成的三线态激子, 以及电激发形成的三线态激子通过RISC过程产生的单线态激子直接以延迟荧光(delayed fluorescence, DF)方式退激, 或者通过Förster能量传递过程(2)实现TBPe的DF. DMAC-DPS器件外量子效率远远超过传统荧光器件外量子效率的理论最大值5%说明了DMAC-DPS的三线态激子有效通过RISC过程形成单线态激子. DMAC-DPS: TBPe器件外量子效率约为基于2-甲基-9,10-二(2-萘基)-蒽(MADN):TBPe器件外量子效率^[17]的3.0倍, 说明了能量有效地通过Förster能量传递过程(2)从包含RISC过程形成部分单线态激子在内的DMAC-DPS单线态激子传递到TBPe. TBPe较低的掺杂浓度抑制了DMAC-DPS同TBPe三线态之间的(3)Dexter能量传递和载流子被TBPe直接俘获(4)等损耗机制的作用.

3 总结

对基于DMAC-DPS的非掺杂型器件进行了优化,

图4 (网络版彩色)发光层分别为15 nm和30 nm的器件*V-I-B*特性(a)和电流密度-效率特性曲线图(b)

Figure 4 (Color online) Characteristics of the devices with 30 nm or 15 nm EML: *V-I-B* plots (a) and current density-efficiency curves (b)

图5 (网络版彩色)DMAC-DPS: TBPe器件的*V-I-B*特性(a), 电流密度-效率特性(b)及DMAC-DPS: TBPe器件与DMAC-DPS器件EL光谱对比图(c)
Figure 5 (Color online) Characteristics of DMAC-DPS: TBPe devices: *V-I-B* plots (a), current density-efficiency curves (b) and electroluminescence spectra of DMAC-DPS: TBPe and DMAC-DPS devices (c)

图6 DMAC-DPS和DMAC-DPS: TBPe器件中激发态的产生和演化
Figure 6 Creation and evolution of excited states in DMAC-DPS and DMAC-DPS: TBPe devices

制备了外量子效率和功率效率分别为14.3%和26.8 lm W^{-1} 的高效蓝光器件. 主要是因为电激发形成的三线态激子有效通过RISC过程到达单线态发光. 在此基础上, 以DMAC-DPS作为主体材料, TBPe作为客体掺杂材料制备了高效的TADF敏化蓝光器件. 该器件的最大外量子效率为12.7%, 约为TBPe的传统荧光器件外量子效率的3.0倍. 这主要是由于在主体材料DMAC-DPS上形成的激子有效的通过Förster能量传递过程传递到TBPe. 分别以TADF材料作为发光材料和主体材料可以制备高效蓝光器件, 这为结构简单高效蓝光器件的制备提供了一种新的途径.

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Summary for “结构简单的高效蓝色荧光器件”

High-efficiency blue fluorescent devices with simple structure

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Thermally activated delayed fluorescence (TADF) materials can harvest both singlet and triplet excitons for light emission without the utilization of heavy metal elements such as iridium and platinum and become the third generation organic optoelectronic materials after fluorescent and phosphorescent materials. TADF material bis[4-(9,9-dimethyl-9,10-dihydroacridine)phenyl]sulfone (DMAC-DPS) shows high photoluminescence quantum yield in solid films, rendering it as a suitable emitter for non-doped organic light emitting diodes. We optimize the hole transport layer, electron transport layer and emission layer thickness for the none-doped DMAC-DPS devices to pursuit high luminance efficiency. We compare the properties of the devices using indium tin oxide (ITO)/MoO₃ hole injection contact with either a 40 nm mCP or 30 nm NPB/10 nm mCP hole transport layer and the results indicate (1) the former devices show larger current than the latter devices for there is a large energy barrier of ca. 0.7 eV for hole injection from NPB into mCP in the latter devices; (2) Both of the devices show similar luminance efficiency and the underlying reason is that the small modulation of the dominant hole current slightly affects the device efficiency. In order to investigate the influence of different electron transport layers on the device performance, we have prepared the devices with either 10 nm DPEPO/40 nm TmPyPB, 50 nm TmPyPB or 50 nm SPPO13 electron transport layer and find out (1) the devices with DPEPO electron transport layer show the highest EQE of 16.2% due to the fact that DPEPO with triplet energy of 3.3 eV can confine excitons inside the emission layer effectively; (2) The devices with SPPO13 possess the highest current density at certain voltage among the devices and the maximum external quantum efficiency and power efficiency for the SPPO13 based devices are 14.3% and 26.8 lm W⁻¹, which are higher than 13.8% and 22.2 lm W⁻¹ for the TmPyPB based devices. The possible reason is that highly polarized phosphorus oxygen groups of SPPO13 greatly improve the electron injection and as a result improve the carrier balance in the devices. Moreover, we compare the properties of the devices with a 15 and 30 nm emission layer and find that the devices with a 30 nm emission layer have slightly better luminance efficiency. High efficiency of the DMAC-DPS devices can be attributed to the effective conversion of triplet excited states to singlet excited states via reverse intersystem crossing (RISC) process. TADF sensitized fluorescence devices can achieve high luminance efficiency by using the well-established fluorescent materials with the advantages such as the superior stability, versatility and abundance. We report highly efficient blue TADF sensitized devices using DMAC-DPS as the host material and the traditional fluorescence blue-emitting material 2,5,8,11-tetra-tert-butylperylene (TBPe) as the guest material. The devices with 1.0% TBPe show the maximum external quantum efficiency and power efficiency of 12.7% and 22.9 lm W⁻¹, which are about 3 times higher than those of the conventional 2-methyl-9,10-bis(naphthalen-2-yl)anthracene (MADN): TPBe devices. This can be attributed to the effective use of triplet excited states for light emission via RISC process and highly efficient Förster energy transfer process from DMAC-DPS to TBPe. Utilization of a TADF material as host material and sensitizer and a traditional fluorescence material as emitting dopant serves as a novel approach to achieve high-efficiency organic light emitting diodes.

thermally activated delayed fluorescence, blue light emitting diodes, sensitization, host materials

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