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PROJECT DESCRIPTION

1. Project Objective:

- To demonstrate high efficiency white emitting OLED devices with a target luminous efficiency between 100 lm/W and 150 lm/W with integrated microcavity structure and down conversion phosphors.
- The main focus of this work will be on three areas:
 - demonstration of a *2X reduction in OLED device operating voltage* by employing the appropriate dopants in the carrier transporting layers
 - demonstration of a *3X light out-coupling efficiency enhancement* by incorporating microcavity structure in the OLED devices
 - demonstration of a *2X down-conversion efficiency enhancement* (from blue to white) using phosphors.

2. Technical Approach and Work Plan:

- Development of n and p doped devices
- Design and fabrication of microcavity devices
- Development of down conversion phosphors
- Device integration

3. Progress-to-Date:

ALL MILESTONES FOR YEAR 3 HAVE BEEN MET.

Summary of deliverables:

- Demonstrated of 50 lm/W blue emitting devices.
- Demonstration of p-conductivity of 10^{-4} S/cm.

Milestone achieved.

Milestone achieved

- Demonstration of OLEDs with an efficiency of 100 lm/W and CRI of 83. **Milestone achieved**

In the following sections, a detail progress report is given.

I. Development of PIN deep-blue FIr6 PHOLEDs

A. Small molecule p-doped hole injection layer

For the p-i-n small molecule OLEDs fabricated in this project, we employed a hole injection layer of MeO-TPD doped with a p-type dopant F₄-TCNQ. The molecular structures of these materials are shown in the inset of Fig. 1. We measured the conductivity of undoped and doped MeO-TPD films using the vertical contact geometry. On a clean glass substrate, a 50 nm thick Au layer was deposited, then a 150-200 nm thick MeO-TPD layer, followed by another 50 nm thick Au layer. The conductivity was obtained from the low voltage region of the current-voltage characteristics of the device, which was linear. For the undoped MeO-TPD film, a conductivity of $(7 \pm 3) \times 10^{-11}$ S/cm was obtained. For doped MeO-TPD films, as shown in Fig. 1, the conductivity increases superlinearly with the F₄-TCNQ doping concentration. With a doping concentration of ~ 10 mol%, the conductivity reaches 1×10^{-4} S/cm, which is more than six orders of magnitude higher than the undoped film.

Fig. 1. Conductivity of MeO-TPD film p-doped with F₄-TCNQ as a function of doping concentration

B. Wide gap hole transporting material

1,1-bis-(di-4-tolylaminophenyl)cyclohexane (TAPC) is used to replace N,N'-Di-[(1-naphthyl)-N,N'-diphenyl]-1,1'-biphenyl-4,4'-diamine (NPD) as the hole transport layer (HTL). TAPC has a triplet energy (T_1) of 2.87 eV, which is nearly the same as N,N'-dicarbazolyl-3,5-benzene (mCP), but significantly greater than that of NPD ($T_1 = 2.29$ eV). Hence, a TAPC layer should provide better exciton confinement in the emissive layer. Fig. 2(a) shows a comparison of the efficiencies of two phosphorescent organic light-emitting devices (PHOLEDs) using different HTLs (NPD vs. TAPC). The device is fabricated with the structure of ITO/ HTLs(40nm)/mCP(10nm)/UGH2:10wt%FIr6(20nm)/BCP(40nm)/LiF(1nm)/Al(50nm). For the TAPC device, η_{EQE} reaches a maximum of $(18 \pm 1)\%$. The maximum power efficiency, achieved at a luminance of $L = 4$ cd/m², is $\eta_P = (16 \pm 1)$ lm/W, which is approximately 15% higher than the maximum of the NPD device (14 lm/W). However, the TAPC device has a $\eta_P = (13 \pm 1)$ lm/W at $L = 100$

cd/m^2 and $\eta_P = (8.9 \pm 0.6) \text{ lm/W}$ at $L = 800 \text{ cd/m}^2$, approximately 60% higher than the NPD device at these two luminances (7.9 lm/W and 5.6 lm/W , respectively).

Fig. 2 (a) Current density-voltage (J - V , closed symbols) and luminance-voltage (L - V , open symbols) characteristics for PHOLEDs with either NPD or TAPC as the hole transport layer. (b) luminous power (η_P , closed symbols) and external quantum (η_{EQE} , open symbols) efficiencies as a function of current density (J) for two PHOLEDs.

C. Optimization of FIr6 doping in p - i - n PHOLEDs

A p - i - n structure composed of ITO/MeO-TPD:F₄-TCNQ(20nm)/TAPC(20nm)/mCP(10nm)/UGH2:FIr6(20nm)/BPhen(20nm)/BPhen:Li(20nm)/LiF(1nm)/Al(50nm) is used to reduce the PHOLEDs drive voltage. Tetrafluoro-tetracyanoquinodimethane (F₄-TCNQ) doped into N, N'-diphenyl-N,N' - bis (3-methylphenyl)- [1,1'-biphenyl]-4,4'-diamine (MeO-TPD) is used as a p-type hole injection layer, and Li doped into 4,7-diphenyl-1,10-phenanthroline (BPhen) is used as an n-type electron injection layer.

Fig. 3(a) shows the L - J - V characteristics of p - i - n S-EML devices with varying iridium(III) bis(4',6'-difluorophenylpyridinato)tetrakis(1-pyrazolyl)borate (FIr6) doping concentration in UGH2, as compared to a conventional S-EML device. The mCP and UGH2 layer thicknesses are 10 nm and 20 nm, respectively, for all these devices. It can be seen that with the same FIr6 concentration (10 wt%) in the EML, the p - i - n device has significantly higher current density than the conventional device at the same voltage. As shown in Fig. 3(b), luminous efficiency (η_L) of these two devices are comparable, with the difference likely due to the different charge balance in these two devices. For the p - i - n devices, as the FIr6 concentration increases, both J and L at a given voltage are significantly increased. Moreover, η_L of the p - i - n devices also increases with the FIr6 doping concentration, from a maximum of $(24 \pm 2) \text{ cd/A}$ for the 10 wt% device to a maximum of $(30 \pm 2) \text{ cd/A}$ for the 25 wt% device. The combination of lower drive voltage and higher η_L in p - i - n devices with higher FIr6 concentrations leads to a maximum $\eta_P = (20 \pm 2) \text{ lm/W}$. The p - i - n devices substantially increase the efficiency at high luminance levels. For example, η_P at 100 cd/m^2 is improved from 13 lm/W for the conventional device to 19 lm/W for the best p - i - n device, up by approximately 50%.

Fig. 3. (a) Luminance (L) – Current density (J) – Voltage (V) characteristics and (b) luminous (η_L) and power (η_P) efficiencies of single-emissive layer (S-EML) p - i - n blue PHOLEDs. The structure for the conventional device is ITO/TAPC(40nm)/mCP(10nm)/UGH2:Fir6(20nm)/BCP(40nm)/LiF(1nm)/Al(50nm), whereas the structure for a p - i - n device is ITO/MeO-TPD:F₄-TCNQ(20nm)/TAPC(20nm)/mCP(10nm)/UGH2:Fir6(20nm)/BPhen(20nm)/BPhen:Li(20nm)/LiF(1nm)/Al(50nm). The Fir6 doping concentration is 10 wt% in the conventional device, whereas it is varied from 10 wt% to 25 wt% in the p - i - n devices.

D. D-EML p - i - n PHOLEDs

Dual emissive layers consisting of an mCP layer doped with 4 wt% Fir6 and a p -bis(triphenylsilyl)benzene (UGH2) layer doped with 25 wt% Fir6 were employed. Instead of a pure mCP layer as an electron/exciton blocking layer (EBL/XBL), Fir6 doped mCP layer maximize exciton generation due to the slight electron leakages and triplet exciton diffusions to the mCP layer from Fir6 in UGH2 layer.

Fig. 4 (a) Current-voltage (J - V) characteristics of single- and dual-emissive-layer (S-EML and D-EML, respectively) p - i - n devices with either 20 nm or 15 nm thick UGH2 layers (doped with 25 wt% Fir6). (b) Power efficiency (η_P) as a function of current density for these S-EML and D-EML p - i - n devices.

As shown in Fig. 4(a), the D-EML devices have slightly higher current densities than the S-EML devices with the same UGH2 thicknesses, mostly due to the slightly thinner mCP layer used (7 nm vs. 10 nm). The D-EML device with a 15 nm thick UGH2:Fir6 layer has a significantly higher η_P than the device with a 20nm thick UGH2:Fir6 layer. This clearly demonstrates the effectiveness of the D-EML structure in harvesting excitons generated in the two emissive layers. The maximum power efficiency of this device is $\eta_P = (25 \pm 2)$ lm/W,

achieved at $L \approx 100 \text{ cd/m}^2$, which is only slightly reduced to $\eta_P = (20 \pm 2) \text{ lm/W}$ at $L = 1,000 \text{ cd/m}^2$.

Table I shows the comparison of the turn-on voltage, efficiencies, and CIE coordinates of different devices. The $p-i-n$ devices not only possess higher peak η_P , but also the luminance corresponding to the peak η_P shifts toward higher luminances, which are much more valuable for use in displays ($L \sim 100 \text{ cd/m}^2$) and lighting ($L \sim 1000 \text{ cd/m}^2$). At these luminances, η_P of the best D-EML $p-i-n$ is more than three times higher than the conventional device with an NPD HTL, or approximately twice or more than that of the conventional device with a TAPC HTL. The EL spectra in Fig. 5(b) do not display appreciable changes among these four devices, and the CIE coordinates of these devices are nearly identical (see Table I).

Device	V_T (V)	η_{EQE} (%) (at peak)	η_L (cd/A) (at peak)	η_P (lm/W) (at peak, 100cd/m ² , 1000cd/m ²)	CIE (x, y) (at 1mA/cm ²)
Conventional (NPD)	4.5 V	12 ± 1	22 ± 2	14, 8, 6 ± 1	(0.16, 0.28)
Conventional (TAPC)	4.1 V	15 ± 1	26 ± 2	18, 13, 8 ± 1	(0.16, 0.27)
S-EML $p-i-n$	3.7 V	15 ± 1	30 ± 2	20, 19, 13 ± 1	(0.17, 0.29)
D-EML $p-i-n$	3.2 V	17 ± 1	35 ± 3	25, 25, 20 ± 2	(0.16, 0.28)

TABLE I. Comparison of the turn-on voltage (V_T), external quantum efficiency (η_{EQE}), luminous efficiency (η_L), power efficiency (η_P), and CIE coordinates of four different devices.

E. High efficiency blue PHOLEDs

The efficiency of an OLED is affected by the charge balance in the emitting layer. Our single carrier device results confirm that the devices are strongly hole dominant leading to triplet exciton quenching by BCP ($T_1 = 2.4 \text{ eV}$) which has lower triplet energy than FIrpic ($T_1 = 2.7 \text{ eV}$). To enhance the charge balance, we used of a high triplet energy, high electron mobility tris[3-(3-pyridyl)-mesityl]borane (3TPYMB) as the ETL in the FIrpic blue PHOLEDs. Hence we fabricated a device replacing 3TPYMB as electron transport layer and compared it to the baseline BCP device. Fig. 5(a) shows the LIV characteristics for the two devices using BCP (device A) and 3TPYMB (device B) as the ETL. As shown in Fig. 5 (b), the device with 3TPYMB show almost 5 times higher current density as compared to BCP devices which clearly shows the effect of high mobility of 3TPYMB on device characteristics.

Fig. 5: a) IV characteristics for devices employing 3TPYMB and BCP as ETL. b) Current efficiency and Power Efficiency comparison for devices employing 3TPYMB and BCP as ETL.

The control device having BCP as an Electron transport layer shows ~ 30 cd/A luminous efficiency and 13 lm/W luminous efficacy. With the use of 3TPYMB as ETL we were able to achieve a high efficiency of 49.4 cd/A at a brightness of 100 cd/m^2 . The peak efficiencies achieved are 49.4 cd/A. As discussed this substantial enhancement in the efficiency is attributed to its high triplet energy and high electron mobility.

Further in devices based on the UGH2 host the transport in the emitting layer is through the dopant molecules. Hence the doping concentration was increased to 15% from 10% in the control device we saw an enhancement in the device performance as shown in Fig. 6.

Fig. 6 Left: Power and current efficiencies of the 15% doped Firpic device. Right: External quantum efficiency of the same device.

F. Microcavity

Blue PHOLED devices with two and four layers of quarter wave stacks (QWS) have been fabricated on the glass substrate. Thicknesses of the dielectric layers are designed to maximize the micro-cavity effect at 475nm. The optimized device has the following structure: glass substrate (1mm)/SiO₂ (79nm) /TiO₂ (48nm) or SiO₂ (79nm)/TiO₂ (48nm)/SiO₂ (79nm)/TiO₂ (48nm)/ITO (50nm)/PEDOT (25nm) /TAPC(20nm) /Firpic:mCP (20nm) /BCP (40nm)/LiF (1nm)/aluminum (100nm). 3% Firpic is doped in emitting layer of mCP host.

Normalized spectra of non-cavity, 2QWS, and 4QWS devices are compared in Fig. 7(a). The spectrum of 4QWS (485nm peak wavelength) device has a narrower FWHM and bluer spectrum than those of the 2QWS and non-cavity devices. The current efficiency and power efficiency of 3 devices are compared in Fig. 7(b). The results of the cavity devices are summarized in Table II. The maximum current efficiency of the 2QWS and 4QWS devices are 32.3 and 30.9 cd/A, respectively. The maximum power efficiency enhancement is 1.88X for the 2QWS device and 1.72X for the 4QWS device. The reason of lower efficiency with the 4QWS device than that of 2QWS device is the dependence of luminance on photopic response. As the spectrum has more blue component, its luminance decreases even the flux increases.

Fig. 7 (a) : Relative spectrum intensity of noncavity, 2QWS, and 4QWS devices

Fig. 7 (b) : Current and power efficiency of noncavity, 2QWS, and 4QWS devices

	Current efficiency (cd/A)	Luminous efficiency (lm/W)
Non-cavity	23.7	10.8
2QWS	32.3 (1.36X)	20.4 (1.88X)
4QWS	30.9 (1.3X)	19.1 (1.72X)

Table II. Comparison of Non-cavity and cavity device performance.

G. Down conversion OLEDs

I. Characterization of down conversion phosphors

A proprietary cerium yellow phosphor along with a red phosphor was tested as down-conversion phosphors. The PL and PLE spectra of the red phosphors were characterized and the peak emission wavelength was at 635 nm, and the CIE color coordinates and the QY of the phosphor were (0.68, 0.32) and 74%, respectively. The particle size distributions were characterized by light scattering and optical microscopy methods. For light scattering analysis, phosphor powders were dispersed in water followed by ultrasonication. The water-dispersed phosphors were drop cast onto a glass substrate and air dried before optical imaging. The PL and PLE spectra, color coordinates and QYs were determined using the JASCO FP6500 spectrophotometer equipped with an integrating sphere, and the properties were suitable for efficient down conversion.

II. Preparation and characterization of Phosphor-Host Thin Films

Phosphor thin films were prepared using two host materials: (1) polymethyl methacrylate, and (2) silicone. The refractive indices of the PMMA and silicone were 1.40 and 1.44, respectively. For PMMA host films, 30mg of phosphor in 500 μ L of PMMA-chlorobenzene solution was used to prepare the films. For silicone hosted thin films, 30 mg of yellow phosphor in 150 mg of silicone was used to prepare the phosphor films.

The optical absorption of the phosphor layer was investigated by transmittance measurements using the JASCO FP-6500. The transmission spectra of both PMMA and silicone host films show a transmission band peaked at 460 nm. The transmission spectra for PMMA-phosphor films exhibit 55-70 % transmission were found across the visible spectrum depending on the phosphor loading, while the transmission of the silicone-phosphor mixtures coated exhibits 60-70% transmission. The low transmission between 400-525 nm is due to absorption of

blue light by the phosphor. The reduced transmission from 525-700 nm is due to scattering by the phosphor particles.

III. Down Conversion using iLED and PMMA-Host Films

Down conversion films were made by drop casting technique. The phosphor films were characterized using blue emitting inorganic (InGaN) LEDs and a custom integrated sphere setup shown in Fig. 8(a). Fig. 8(b) shows the CIE coordinates of the photoluminescence (PL) spectra for four samples with different amount of phosphors in PMMA/chlorobenzene solution. Normalized PL spectra of four phosphor samples along with the LED emission spectrum are shown in Fig. 8(c). The relative external efficacy of three identical down conversion samples, prepared using 500 μL of a 60 mg/mL solution, are shown in Fig. 8(d) and *all three samples showed a 2X increased efficacy due to down conversion from blue to white light.*

Fig. 8: (a) Customized setup for LED-phosphor down-conversion experiments; (b) CIE diagram coordinates for phosphor, blue LED and four samples with PMMA-host down conversion layer; (c) Normalized PL spectra from the four down conversion samples, including spectra from the blue LED and from the phosphor; (d) Relative efficacy vs. LED input power for three identical samples and blank blue LED showing a factor of 2X increase.

IV. Down-Conversion using iLED and Silicone-Host Sample

Down-conversion properties of blue to white light by using a silicone host for the yellow phosphor was also tested. The normalized down converted PL spectra from 20mg, 30mg, 40mg and 50mg of phosphor in 150mg of silicone are shown in Fig. 9(a). The relative efficacy of the LED device and down converted spectra was determined. *Again note that the relative efficacy values of down converted white light (Fig. 9 (b)) are 2X higher than the original blue emission,*

meeting our milestone. The CRI values obtained were 76, 76, 64, and 60 for the 20, 30, 40 and 50 mg samples, respectively.

Fig. 9(a): Normalized down converted spectra from iLED/phosphor/silicone-host layer, plus the pure blue emission spectrum from the iLED; (b) Relative efficacy vs. LED input power for four different phosphor loading and blank blue iLED showing a factor of 2X increase.

V. Down Conversion using OLED Device and Phosphor Thin Films

Blue emitting organic light emitting devices (OLEDs) with and without a microcavity were integrated with a phosphor coated glass slides and tested for down-conversion to white light. The down conversion layer consisted of 30 mg of the yellow phosphor mixed with 150 mg of silicone. In another down conversion film, 22.5g of yellow phosphor and 7.5 g of red phosphors were mixed with 150 mg of silicone. Down conversion spectra from OLEDs are shown in Fig. 10 for 30 mg of yellow phosphor or a 3:1 mixture of yellow:red phosphors in 150 mg of silicone. The CRI values obtained were 66 and 89 for the single yellow and red-yellow mixture, respectively.

Fig. 10: Emission spectra from pure OLED and down converted blue light using either a single yellow or dual red:yellow phosphors.

VI. Fabrication of down conversion white OLEDs

Micro-cavity blue PHOLEDs were fabricated on the two different dielectric mirrors: two-layer quarter wave stacks with reflectivity of 0.39 ($R_{2QWS} = 0.39$), and 4-layer quarter wave stacks with reflectivity of 0.7 at 475nm ($R_{4QWS} = 0.7$). The thickness of each dielectric layer is designed to maximize the micro-cavity effect at 475nm by the optical OLED simulation[79]. The devices have the following structure: glass substrate (1mm)/SiO₂ (79nm)/TiO₂ (48nm) or SiO₂ (79nm)/TiO₂ (48nm)/SiO₂ (79nm)/TiO₂ (48nm)/ITO (50nm)/ PEDOT:PSS (60nm)/ TAPC (25nm)/ mCP (25nm) with 20 wt % FIrpic/ 3TPYMB (40nm)/ CsCO₃(0.8nm)/ aluminum (100 nm) in figure 5.6. The thickness of the PEDOT:PSS layer cautiously was optimized to achieve the maximum resonant cavity effect.

The current efficiency and power efficiency without down conversion phosphor films are shown in Figure 5.7(a). The maximum current and luminous power efficiencies of noncavity, 2QWS, and 4QWS OLEDs was 44.3, 45.7, 43.8 cd/A and 39.7, 39.9, 41.1 lm/W. The spectra of 2QWS and 4QWS devices become narrow and more bluish and the power efficiency of both the noncavity and microcavity devices are almost the same. However the EQE of the microcavity devices is ~20% higher than that of noncavity device due to blue shifted spectrum. Three different thicknesses of yellow and yellow: red phosphor mixture films were prepared on the separate glass slides. The phosphor films were applied to the blue micro-cavity PHOLED substrate with an index matching gel to minimize the total internal reflection (TIR). Finally, a macrolens with index matching gel was applied on the top of down conversion phosphor film to increase light out-coupling efficiency with index matching gel.

VII. High efficiencies of down conversion white OLEDs

The current efficiency and power efficiency of integrated down conversion WOLEDs are summarized in Table IV. The efficiency enhancements using the macrolens are 46% and 31% for yellow and yellow: red mixture phosphor films, respectively. The power efficiencies of the down conversion OLEDs with 4QWS microcavity show efficiencies of 61 lm/W for the Y180 phosphor device and 57 lm/W for the YR180 phosphor device at 100 cd/m².

This efficiency enhancement due to the microcavity device is attributed to 3 reasons. First, the blue shifted spectrum of microcavity device is a better match to the phosphor excitation spectrum than the noncavity device. Second, the external quantum efficiency of the cavity device is higher. Third, there is a stronger substrate guided mode in the microcavity device. This substrate guided mode is utilized and harvested due to phosphor powder scattering. It is known that phosphor films increases the light extraction efficiency from 10-40% depending on the concentration and thickness of phosphor film.

To further enhance the efficiency, a macrolens was used to extract the substrate guided modes in phosphor films. Efficiency enhancements due to the macrolens are 42% and 31% for the yellow phosphor and the yellow: red mixture phosphor devices respectively. *The resulting power efficiencies of the down conversion OLEDs are 107 lm/W (CRI : 59) and 99.3 lm/W (CRI : 83) for the yellow phosphor and the mixture of red: yellow phosphors at 30 cd/m².*

Label	Silicone A (mg)	Silicone B (mg)	Yellow Phosphor (mg)	Red Phosphor (mg)	Total Phosphor Wt (mg)	Film Thickness (μm)
Y90	30	30	30	--	90	95
YR90	30	30	20	10	90	-
Y180	75	75	30	--	180	220
YR180	75	75	20	10	180	-
Y360	165	165	30	--	360	450
YR360	165	165	20	10	360	-

Table III. Weight of components in different down-conversion thin film samples

	cd/A (30, 100, 1000 cd/m ²)	lm/W (30, 100, 1000 cd/m ²)	CIE (x, y)	CRI
Noncavity	39.9, 43, 30.5	38, 38, 21	(0.15, 0.32)	-
Noncavity + Y180	50, 45, 29	46, 37, 18	(0.34, 0.52)	55
Noncavity + Y180 + Macrolens	65, 59, 38	59, 51, 26	(0.34, 0.52)	55
Noncavity + YR 180	46, 40, 20	41.6, 32, 16	(0.42, 0.47)	80
Noncavity + YR 180 + Macrolens	60, 54, 35	54, 45, 23	(0.42, 0.47)	80
4QWS	42, 39, 26	41, 34, 18.7	(0.15, 0.22)	-
4QWS + Y180	75, 70, 45	73, 61, 32	(0.36, 0.50)	59
4QWS + Y180 + Macrolens	109, 106, 71	107, 96, 53	(0.36, 0.50)	59
4QWS + YR180	69, 64, 41	67.7, 57, 30	(0.43, 0.46)	83
4QWS + YR180 + Macrolens	101, 93, 60	99.3, 87, 47	(0.43, 0.46)	83

Table IV. Summary of current and power efficiencies for down conversion of noncavity and 4QWS microcavity OLEDs at 30, 100, and 1000 cd/m². CIE and CRI are also shown here.

4. Milestones/Deliverables:

All milestones in Year 3 have been met:

- Demonstrated 50 lm/W blue emitting devices *without using microcavity*
 - Achieved high efficiency by carefully balancing the charge transport, **exceeding milestone target.**
- Demonstrated PIN OLED devices
- Demonstrated light out-coupling enhancement due to microcavity structure.
- Demonstrated down conversion OLEDs with a maximum power efficiency of 100 lm/W and a CRI of 83.