Charge conduction study of phosphorescent iridium compounds for organic light-emitting diodes application

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Abstract:

The charge conduction properties of a series of iridium-based compounds for phosphorescent organic light-emitting diodes (OLEDs) have been investigated by thin-film transistor (TFT) technique. These compounds include four homoleptic compounds: $Ir(ppy)_3$, $Ir(piq)_3$, $Ir(Tpa-py)_3$, $Ir(Cz-py)_3$, and two heteroleptic compounds $Ir(Cz-py)_2(acac)$ and FIrpic. $Ir(ppy)_3$, $Ir(piq)_3$ and FIrpic are commercially available compounds, while $Ir(Tpa-py)_3$, $Ir(Cz-py)_3$ and $Ir(Cz-py)_2(acac)$ are specially designed to test their conductivities with respect to the commercial compounds. In neat films, with the exception of FIrpic, all Ir-compounds possess significant hole transporting capabilities, with hole mobilities in the range of about 5×10^{-6} to 2×10^{-5} cm^2 $V^{-1}s^{-1}$. FIrpic, however, is non-conducting as revealed by TFT measurements. We further investigate how Ir-compounds modify carrier transport as dopants when they are doped into a phosphorescent host material CBP. The commercial compounds are chosen for the investigation. Small amounts of $Ir(ppy)_3$ and $Ir(piq)_3$ (< 10%) behave as hole traps when they are doped into CBP. The hole conduction of the doped CBP films can be reduced by as much as 4 orders of magnitude. Percolating conduction of Ir-compounds occurs when the doping concentrations of the Ir-compounds exceed 10%, and the hole mobilities gradually increase as their values reach those of the neat Ir films. In contrast to $Ir(ppy)_3$ and $Ir(piq)_3$, FIrpic does not participate in hole conduction when it is doped into CBP. The hole mobility decreases monotonically as the concentration of FIrpic increases due to the increase of the average charge hopping distance in CBP.

1. Introduction

Organic light-emitting diodes (OLEDs) are now firmly established as key ingredients in high brightness, sharp contrast, ultra-thin, and light-weight displays [1, 2]. Commercial products are now available in micro-displays, cell phones, tablets, and large area TV sets. Despite these successes, continuous efforts are still needed in more demanding applications such as in solid-state lighting applications. Among these efforts, the design of high efficiency phosphorescent light-emitters takes the central stage. Ir(III) phosphors can capture energies from both singlet and triplet excited states, lifting the limit of the internal quantum efficiency to nearly 100% [3]. Besides light-emitting functions, improving charge transporting properties of Ir(III) phosphors via tailoring their structures can be taken as a useful strategy to optimize the OLED efficiency. The incorporation of bulky and hole-transporting arylamine group or carbazole group can reduce the barrier height of hole-injection and enhance the hole-transporting abilities of the resulting Ir(III) phosphors [4]. In a previous report, we employed thin film transistor (TFT) to probe the conductivity of an archetype metal-based phosphorescent compound, tris(2-phenylpyridine)iridium(III) [Ir(ppy)$_3$] [5]. We showed that Ir(ppy)$_3$ is hole conducting, and its hole mobility is of the order of 2×10^{-5} cm^2 V^{-1}s^{-1}, which is comparable to a common hole transporter 4,4'-bis[*N*-(1-naphthyl)-*N*-phenylamino]biphenyl (NPB) under similar measuring conditions. As Ir(ppy)$_3$ possesses both light-emitting and hole transporting capabilities, incorporating this material into an OLED should allow for device simplification [6-13]. One example of such an effort is to use a neat film of Ir(ppy)$_3$ as a hole injection layer in an OLED [6]. A more appealing approach is to use Ir(ppy)$_3$ as a dopant to modulate the hole injection and conduction properties of a wide band gap hole transporter. Lui et al. reported a high efficiency and simplified OLED that uses the Ir(ppy)$_3$

responsible for hole injection from ITO anode and hole transport in the emissive layer (EML) of CBP [6-7]. Later on, Ma et al. and Kim et al. also demonstrated that the phosphorescent dopants, Ir(ppy)$_3$ and Ir(ppy)$_2$acac, can contribute to the charge transport in EML in OLED, which can reduce the barrier at the interface of HTL and EML, or even replace the hole transporting layer (HTL), such as TAPC and NPB [11-13]. With this approach, simplified OLEDs are demonstrated to have significantly enhanced operational stability without loss of efficiency [13]. To further develop this kind of strategy for device simplification, it is therefore necessary to have more in depth understanding of the charge conduction properties in metal-based phosphorescent compounds.

This contribution looks into the hole conduction in a series of Ir-based phosphorescent compounds that have been applied in OLEDs. They include homoleptic Ir(ppy)$_3$, Ir(piq)$_3$, Ir(Tpa-py)$_3$, Ir(Cz-py)$_3$, and heteroleptic Ir(Cz-py)$_2$(acac) and FIrpic. The chemical structures of all compounds are shown in Fig. 1. Ir(ppy)$_3$, Ir(piq)$_3$ and FIrpic are commercially available compounds, while Ir(Tpa-py)$_3$, Ir(Cz-py)$_3$ and Ir(Cz-py)$_2$(acac) are specially designed to test their conductivities with respect to the commercial compounds. Ir(ppy)$_3$ and Ir(piq)$_3$ employ phenylpyridines as the hole conducting units. Ir(Cz-py)$_3$ and Ir(Cz-py)$_2$(acac) possess a carbazole moiety while Ir(Tpa-py)$_3$ a triphenylamine moiety. Hole mobilities of these compounds were evaluated by TFT technique. In addition, we selected three of the commercial materials, namely, Ir(ppy)$_3$, Ir(piq)$_3$ and FIrpic, and doped these Ir compounds into 4,4'-*N,N'*-dicarbazole-biphenyl (CBP), which is a popular charge transporting host used in phosphorescent OLEDs [14], and observed how the hole mobilities change as the dopant concentration varies.

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2. Experimental details

Ir(ppy)$_3$, Ir(piq)$_3$ and FIrpic were obtained from Lumtec Inc. and were used as received. Ir(Tpa-py)$_3$, Ir(Cz-py)$_3$ and Ir(Cz-py)$_2$(acac) were synthesized and purified according to the procedures outlined in the literature [15-17]. TFT technique (top-contact configuration) was used to study the hole transporting properties of the neat or doped CBP films [18, 19]. A thermally grown SiO$_2$ (~300 nm) on a p^+ Si wafer was used as the substrate. The substrate was cleaned by de-ionized water and acetone followed by UV ozone treatment [20]. Then 100 nm of CBP neat film and doped CBP films were thermally evaporated onto the SiO$_2$ layer at a base pressure of 10^{-6} torr. The deposition rate was controlled at 0.5Å/s. Following the organic film, the patterned source (S) and drain (D) electrodes consisted of a thin layer (20 nm) of MoO$_x$ followed by 100 nm of gold thermally deposited through a shadow mask with channel length (L) and width (W) defined by 50 µm and 9 mm, respectively.

3. Results and discussions

Fig. 2 shows the TFT output characteristics of the neat films of CBP, Ir(ppy)$_3$ Ir(piq)$_3$, Ir(Tpa-py)$_3$, Ir(Cz-py)$_3$ and Ir(Cz-py)$_2$(acac) films at room temperature (292 K). They all have well-defined p-type TFT current-voltage characteristics. A clear linear region and saturation region allow us to apply the following standard expressions to evaluate the hole mobilities [21].

$$I_D = \frac{W}{L} \mu_{LIN} C_i \left[V_D (V_G - V_T) - \frac{V_D^2}{2} \right] \qquad \text{Eq. (1)}$$

$$I_D = \frac{W}{2L} \mu_{SAT} C_i (V_G - V_T)^2 \qquad \text{Eq. (2)}$$

where W (= 9 mm) and L (= 50 µm) are the channel width and length, respectively; C_i is the capacitance of gate dielectric per unit area; V_T is the threshold voltage; V_G and

V_D are the gate bias and the potential difference across the S/D electrodes, respectively; μ_{LIN} and μ_{SAT} are the field effect (FE) mobility extracted from the linear and saturation regions, respectively. For CBP, the μ_{LIN} and μ_{SAT} were, respectively, found to be $1.2 \times 10^{-5} \, cm^2 \, V^{-1}s^{-1}$ and $1.5 \times 10^{-5} \, cm^2 \, V^{-1}s^{-1}$, which is comparable to a widely used amorphous organic hole transporter NPB ($\mu_{SAT} = 2.4 \times 10^{-5} \, cm^2 \, V^{-1}s^{-1}$) measured with the same technique [19].

With the exception of FIrpic, all neat films of the iridium compounds show significant hole transporting capabilities. Table 1 shows a summary of the mobility results and their HOMO energy levels. The mobilities vary in the range of 0.5×10^{-5} $cm^2 \, V^{-1}s^{-1}$ to $2 \times 10^{-5} \, cm^2 \, V^{-1}s^{-1}$. Charge conduction in iridium compounds are controlled by both the Ir(III) metal atom and the ligands [22, 23]. Besides the 2-phenylpyridine skeleton, carbazole and arylamine should be very good candidates for the hole-transporting enhancement of iridium(III) complexes [24]. Many carbazole moieties are known to transport holes in organic devices, e.g., poly(9-vinylcarbazole) (PVK) is a recognized hole transporter while CBP is described as a more bipolar transporter. As indicated in Fig. 1(d) and (e), we combined the 9-phenylcarbazole unit with the 2-phenylpyridine ligand to synthesize the iridium(III) complexes Ir(Cz-py)$_3$ and Ir(Cz-py)$_2$(acac), according to the previous report [15]. In addition, arylamine units have been extensively used as hole-transporting functional groups in photoelectronic materials, attributable to their electron-donating nitrogen atom [25, 26]. As indicated in Fig. 1(f), diphenylamine was attached to the phenyl ring of Ir(ppy)$_3$ to obtain Ir(Tpa-py)$_3$ following the synthesis method of the published method [17]. In both homolytic Ir(Cz-py)$_3$ and Ir(Tpa-py)$_3$, their hole mobilities are very similar to Ir(ppy)$_3$ and Ir(piq)$_3$, with saturation TFT mobilities between $1-2 \times 10^{-5} \, cm^2$

$V^{-1}s^{-1}$ (Table 1). However, for the heterolytic Ir(Cz-py)$_2$(acac), its hole mobility is distinctly lower, and has a value of 5×10^{-6} cm^2 V^{-1}s^{-1}. The origin of lower hole mobility for Ir(Cz-py)2(acac) is not clearly known. One possibility is that it has stronger ligand field effect than the C-N ligand [27, 28]. The stronger ligand field causes a red-shift of emission spectrum, and may also work as hole trap in the device.

We have also taken the OTFT measurement on a neat FIrpic film. However, FIrpic shows negligible hole conduction. There is no observable field effect behavior in the measurement. We believe that the poor hole conducting property is mainly due to the absence of an electron donating unit within the molecule. In each FIrpic molecule, the four F atoms behave as electron acceptors rather than donors. So FIrpic is unfavorable for hole transport. The strongly electron-withdrawing fluorine (F) atoms on the phenyl ring of difluorophenylpyridine (dfppy) ligand increase the oxidation potential, resulting in a much lower HOMO energy level (5.80 eV), which inhibits the injection and transportation of hole [29-31].

In phosphorescent OLEDs, Ir(ppy)$_3$, Ir(piq)$_3$ and FIrpic are the widely used dopants in CBP. So it is interesting to study the transport properties of CBP films doped with Ir(ppy)$_3$, Ir(piq)$_3$ and FIrpic, respectively. Fig. 3 shows, as examples, the effects of the three dopants at a doping concentration of 20% inside CBP. For comparison, we also show the transfer characteristics of the neat films of CBP, Ir(ppy)$_3$ and Ir(piq)$_3$. Transfer characteristics of FIrpic is not available due to its poor hole mobility. Compared to the neat CBP, Ir(ppy)$_3$ and Ir(piq)$_3$ films, the output currents I_D of 20% Ir(ppy)$_3$ and Ir(piq)$_3$ doped in CBP films are substantially reduced. They both have the same FET hole mobility $\mu_{SAT} = 2.0 \times 10^{-7}$ cm^2 V^{-1}s^{-1}, which are two orders of magnitude smaller than the neat film of CBP (Table 1). On the other hand, the reduction of the FIrpic doped film is only one order less. The FET hole

mobility is $\mu_{SAT} = 2.0 \times 10^{-6}\,\mathrm{cm^2\,V^{-1}s^{-1}}$ and it is much higher than the other two doped films.

To understand this phenomenon, we look into the HOMO levels of the CBP host and the Ir compounds as shown in Table 1. We note that the HOMO levels of Ir(ppy)$_3$ (5.4 eV) and Ir(piq)$_3$ (5.1 eV) are much smaller than that of CBP (6.0 eV). So Ir(ppy)$_3$ and Ir(piq)$_3$ molecules should be the preferential hole residing sites when they are mixed with CBP. In contrast, the HOMO energy for FIrpic (5.8 eV) is comparable to the CBP host. So there is no preference for holes to reside on FIrpic molecules. In fact, in a FIrpic doped CBP film, injected holes may even prefer to transport in CBP molecules as these molecules form a connected network with a much higher hole mobility. We have investigated the DFT and TD-DFT data of Ir(Cz-py)$_3$ and Ir(Czppy)$_2$acac, and thus found that the metal dπ orbitals' contribution to the HOMOs are 24.5% [16] and 37.9% [22], respectively, which are smaller than other kinds of iridium(III) complexes [23]. So, we think that the hole-transporting nature of carbazole group enhances the conduction of holes.

Besides 20% doping in CBP, we have also attempted a wide range of doping concentrations, e.g. 0%, 0.5%, 10%, 20%, 50% and 100%. Figures 4 and 5, respectively, show the transfer characteristics and the FET mobilities of the films of CBP:Ir(ppy)$_3$, CBP:Ir(piq)$_3$ and CBP:FIrpic in various doping concentrations. For the cases of CBP:Ir(ppy)$_3$ and CBP:Ir(piq)$_3$, they have similar trends and FET mobilities. As indicated in Fig. 5, for a low doping level (<10%), the current and the FET mobility decrease rapidly with the doping concentration. The sharp drop is attributed to the trapping effect of dopants in CBP. At 10% of doping concentration, the output TFT signals were barely observable and no reliable signal and mobility values can be obtained at room temperature. The TFT signals can only be observed at higher

temperature (~390 K). So the mobility values at 292 K were evaluated from higher temperatures at this doping concentration. At a higher doping level (>10%), the output current and mobilities increase with the doping concentration because hopping among the dopants [Ir(ppy)$_3$ or Ir(piq)$_3$] is allowed as the average hopping distance decreases at a higher doping level.

In contrast to CBP:Ir(ppy)$_3$ and CBP:Ir(piq)$_3$, the hole mobility of CBP:FIrpic shows a different concentration dependence as shown in Fig. 5 (triangle symbols). The output current and FET mobility decreases monotonically with the increase in doping concentration. In this case, CBP dominates the hole transport since FIrpic has a negligible hole mobility (extremely poor hole transport property). At a high doping concentration of FIrpic, the average hole hopping distance between adjacent CBP molecules increases and results in a smaller output current and hole mobility.

To check if the hopping distance plays a key role in the conduction of the doped CBP films, we performed the temperature dependence experiment. We employ the Gaussian disorder model (GDM) to analyze our results [32]. In GDM, an amorphous organic material is treated as an ensemble of charge transporting sites for hopping conduction. The carrier mobility is electric field and temperature dependent. It can be described as follows [32]:

$$\mu(F,T) = \mu_\infty \exp\left[-\left(\frac{2\sigma}{3kT}\right)^2\right]\exp\left\{CF^{\frac{1}{2}}\left[\left(\frac{\sigma}{kT}\right)^2 - \Sigma^2\right]\right\} \qquad \text{Eq. (3)}$$

F, T, and k are the applied electric field, the absolute temperature and the Boltzmann constant, respectively; μ_∞ is the high temperature limit of mobility, and C is a constant. The energetic disorder σ can be interpreted as the width of Gaussian distribution of DOS of energy for the charge transporting sites; the positional disorder Σ describes the geometric randomness of the material. At low field ($F \rightarrow 0$), the second

exponential term in Eq. (3) approaches 1, and thus, $\mu(0,T) \sim \exp[-4/9(\sigma/kT)^2]$. A semi-log plot of low field mobilities (using μ_{LIN}) versus $1/T^2$ should yield a straight line. σ and μ_∞ can be extracted from the slope and y-intercept of the line, respectively. The high temperature limit of mobility, μ_∞, can be taken as a measure of the mean hopping distance. Following the concept of the GDM, μ_∞ is directly proportional to the hopping rate across the neighboring charge transporting sites. The rate can be estimated by [33]:

$$\mu_\infty \approx Aa^2 \exp(-\gamma a) \hspace{4cm} \text{Eq. (4)}$$

where γ is the inverse localization radius, a is the hopping distance and A is a prefactor that can be affected by the phonon frequencies. As γ is primarily determined by the molecular structure, it is reasonable to assume that it is a constant. Therefore, μ_∞ can be taken as a measure of the mean hopping distance in the neat and doped films of CBP.

Fig. 6 shows μ_{LIN} vs $(1/T)^2$ for the neat film of CBP and 20% doped CBP films. For the neat film of CBP, $\mu_\infty \approx 6 \times 10^{-3}$ cm^2 V^{-1}s^{-1}. For CBP:Ir(ppy)$_3$ and CBP:Ir(piq)$_3$ at 20% doping level, the extracted μ_∞ are substantially reduced by more than two orders of magnitude to 5.0×10^{-5} cm^2 V^{-1}s^{-1} and 2.5×10^{-5} cm^2 V^{-1}s^{-1}. In these two cases, the dopants control the hole conduction, and it is energetically favorable for holes to hop in-between the dopants. But the mean hopping distances, estimated from Eq. (4) are $a = 1.7$nm for CBP:Ir(ppy)$_3$ and $a = 1.8$ for CBP:Ir(piq)$_3$, respectively. They are much larger than the neat films of CBP, e.g. $a = 0.9$nm. However, for CBP:FIrpic, $\mu_\infty \approx 7 \times 10^{-3}$ cm^2 V^{-1}s^{-1}, and there is no clear difference in μ_∞, in comparison to a neat film of CBP. In this case, the CBP host controls the hole transport, and the FIrpic has little impact on the mean hopping distance. Table 2

summarizes the transport parameters of CBP and doped CBP extracted by GDM model using data from Fig. 6.

4. Conclusion

We study the hole transporting properties of phosphorescent iridium-based molecules in neat films and as dopants in a CBP host. All iridium compounds bearing phenylpyridine, carbazole, and amine moieties show significant hole transporting capabilities, with the FE hole mobilities of $\sim 1 \times 10^{-5}$ cm^2 V^{-1}s^{-1}, comparable to CBP or NPB. FIrpic shows a negligible hole transporting property. In doped films of CBP:Ir(ppy)$_3$ and CBP:Ir(piq)$_3$, dopants act as deep trapping sites at low concentrations due to a relatively low HOMO level. For CBP:FIrpic, hole mobility decreases with the doping concentration (due to a negligible hole transport capability of FIrpic). Conduction in phosphorescent dyes appears to be a general phenomenon, and should be fully explored to simplify OLED structures.
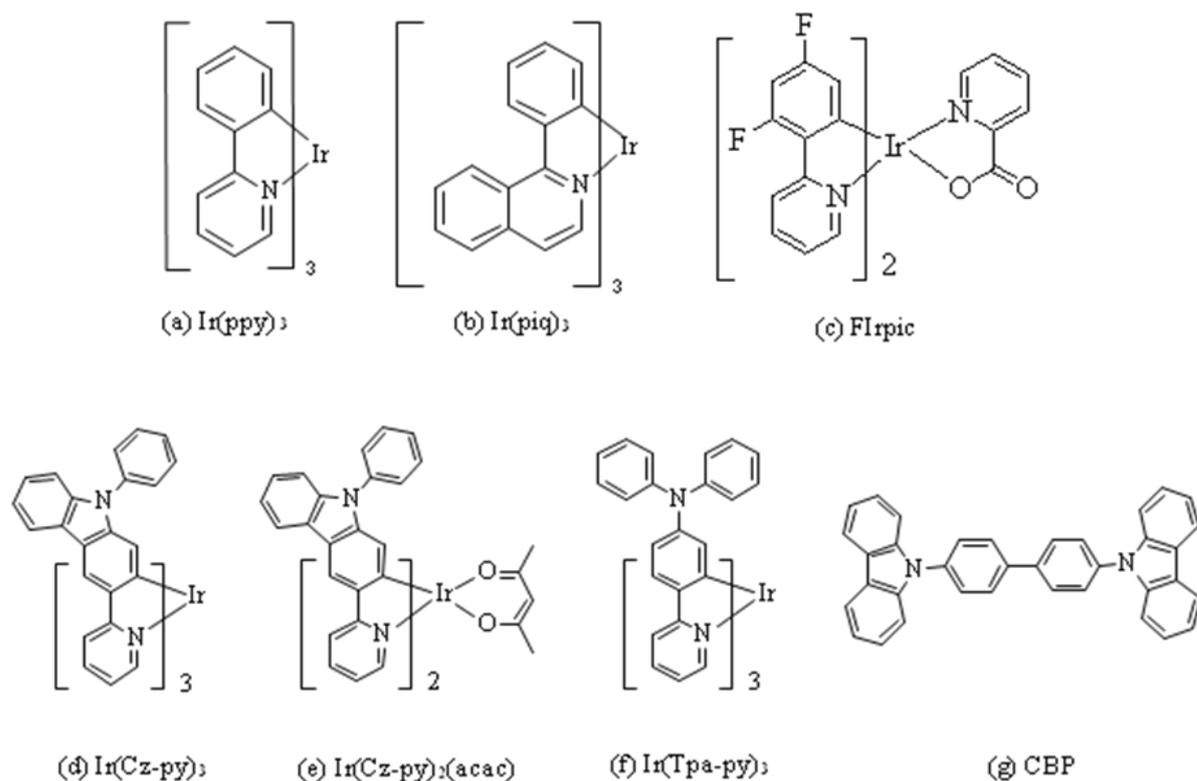
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(a) Ir(ppy)₃   (b) Ir(piq)₃   (c) FIrpic

(d) Ir(Cz-py)₃   (e) Ir(Cz-py)₂(acac)   (f) Ir(Tpa-py)₃   (g) CBP

a) Ir(ppy)$_3$: Tris[2-phenylpyridinato-C2,N]iridium(III)
b) Ir(piq)$_3$: Tris[1-phenylisoquinolinato-C2,N]iridium(III)
c) FIrpic   : Bis[2-(4,6-difluorophenyl)pyridinato-C2,N] (picolinato) iridium(III)
d) Ir(Cz-py)$_3$: Tris{2-[3-(*N*-phenylcarbazolyl)]pyridinato-C2,N}iridium(III)
e) Ir(Cz-py)$_2$(acac) : Iridium(III) bis[2-(9-phenylcarbazolyl) pyridinato-C2,N]
                          acetylacetonate
f) Ir(Tpa-py)$_3$: Tris[2-(triphenylamine)pyridinato-C2,N]iridium(III)
g) CBP   : 4,4-Bis(N-carbazolyl)-1,1'-biphenyl

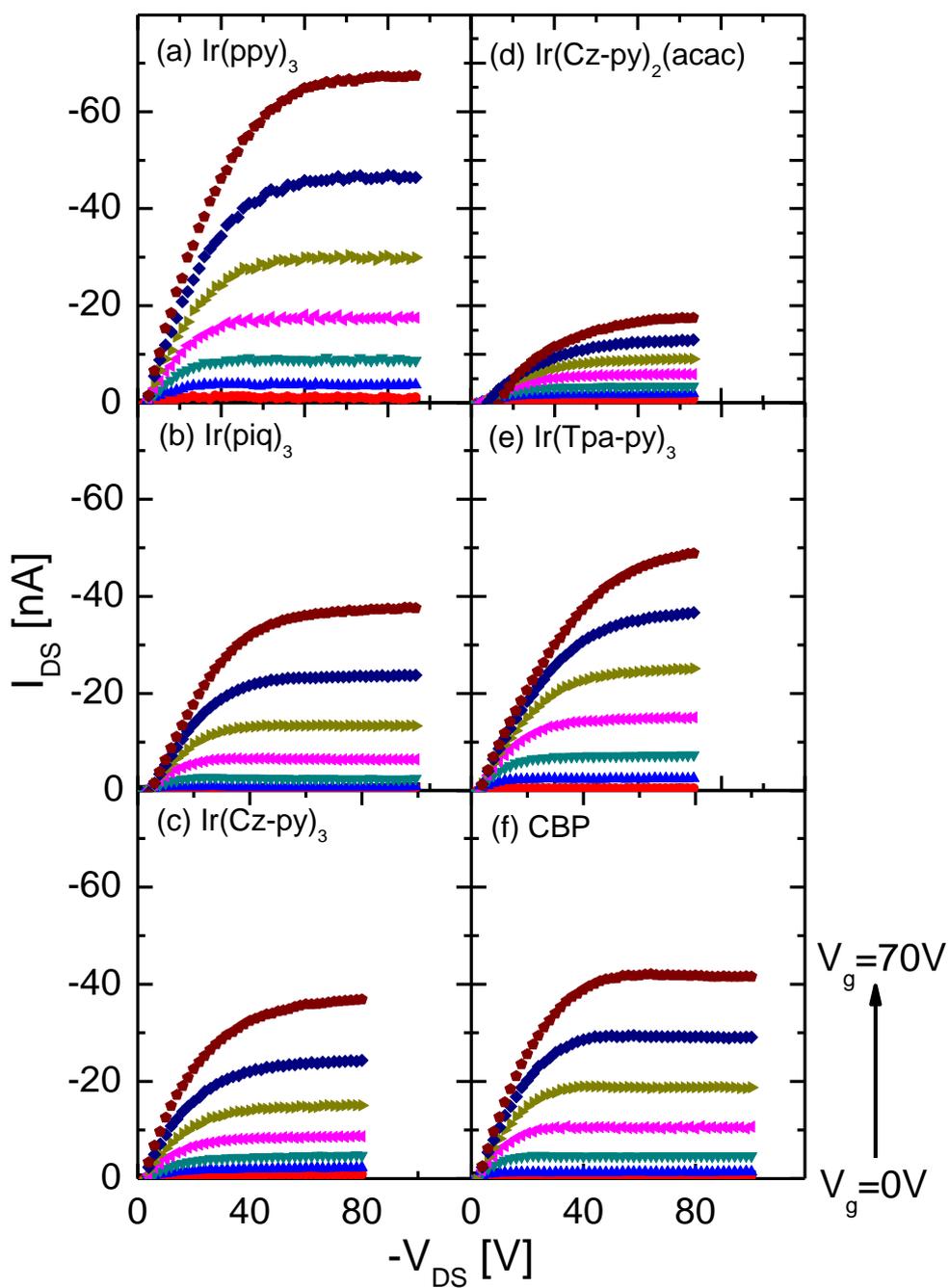Fig. 1. Full names and chemical structures of Ir compounds for conductivity investigation.

Fig. 2. TFT output characteristics of (a) $Ir(ppy)_3$, (b)$Ir(piq)_3$, (c) $Ir(Cz-py)_3$, (d) $Ir(Cz-py)_2(acac)$, (e) $Ir(Tpa-py)_3$, and (f) CBP. The gate voltage increases in step of 10 V.
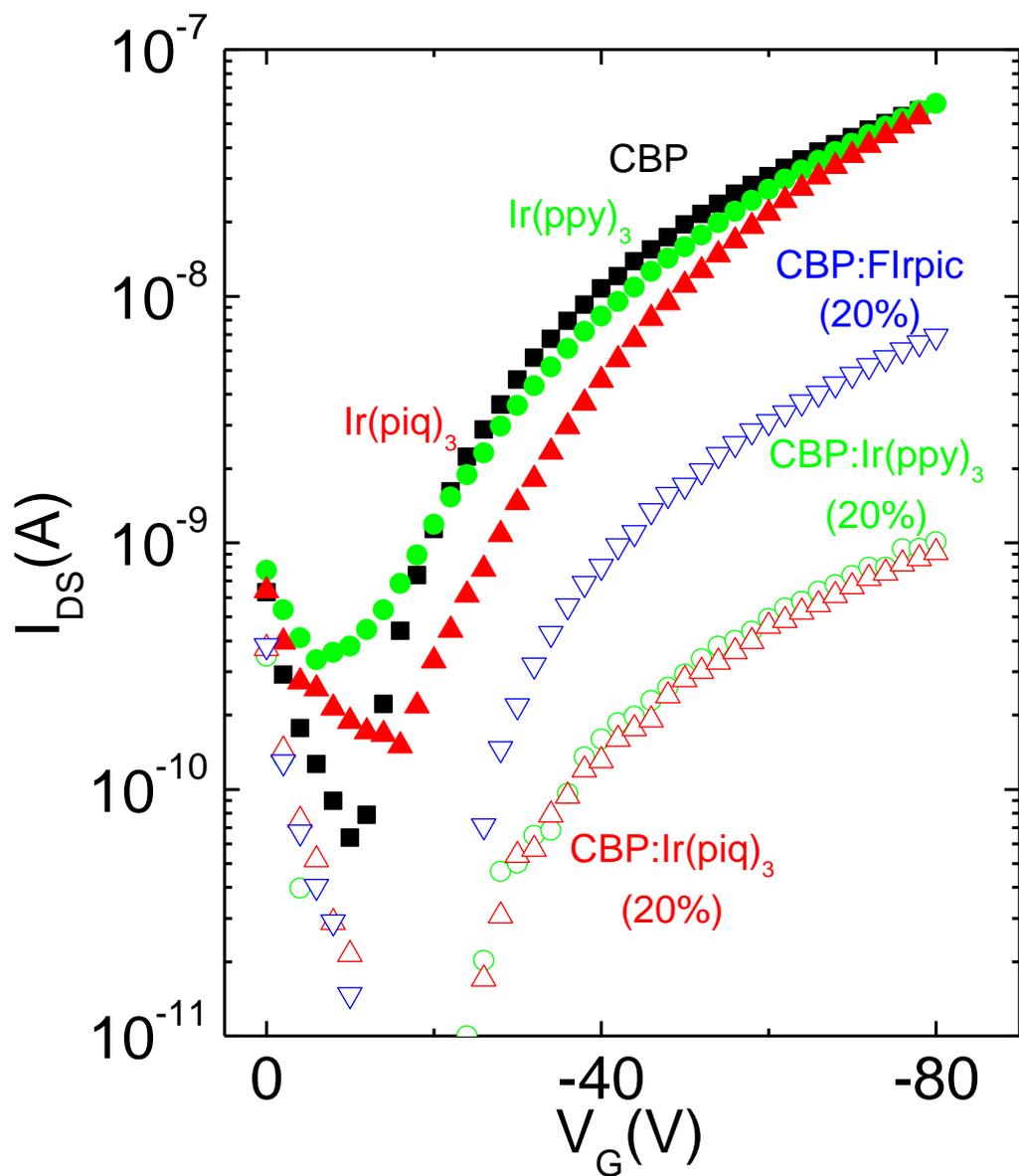
Fig. 3. Transfer characteristics of neat films (solid symbols) and doped films (open symbols) of CBP, Ir(ppy)$_3$ and Ir(piq)$_3$. The doping concentration is 20%.
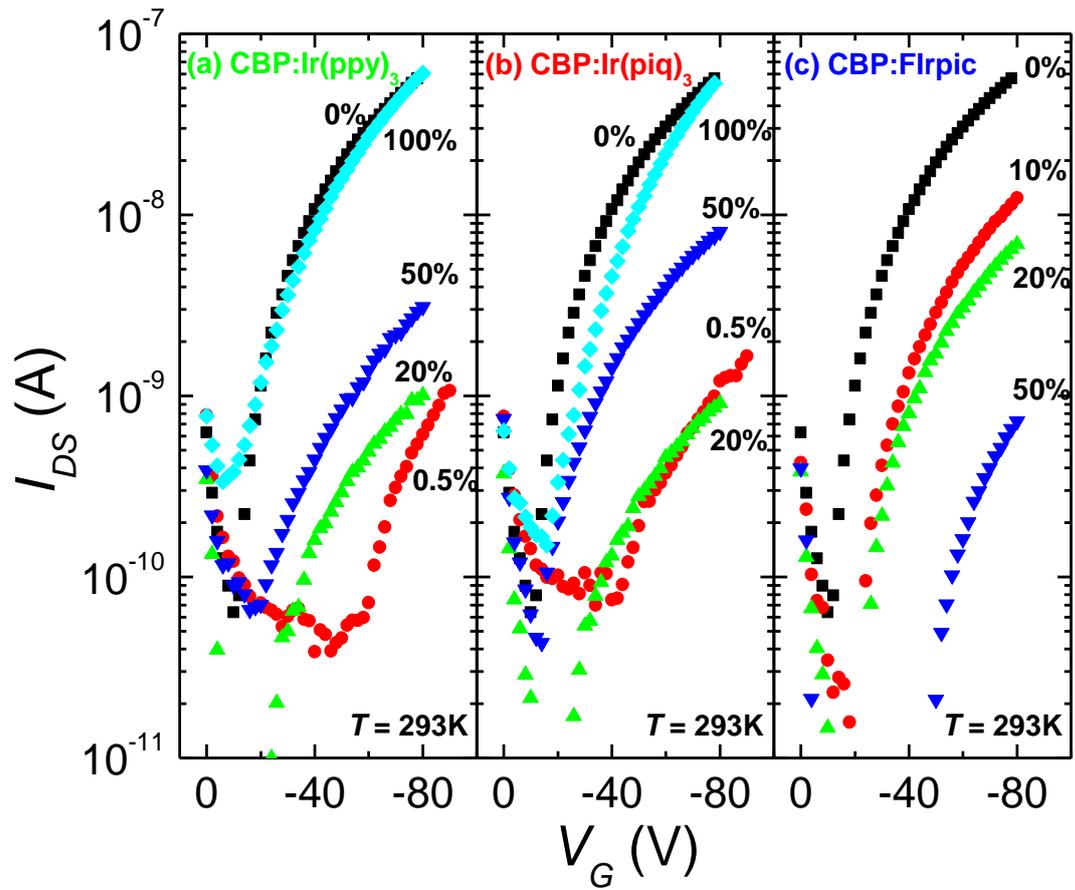
Fig. 4. Transfer characteristics of doped films of CBP using (a) Ir(ppy)$_3$, (b) Ir(piq)$_3$ and (c) FIrpic as dopants. The doping concentration varies from 0 to 50%.
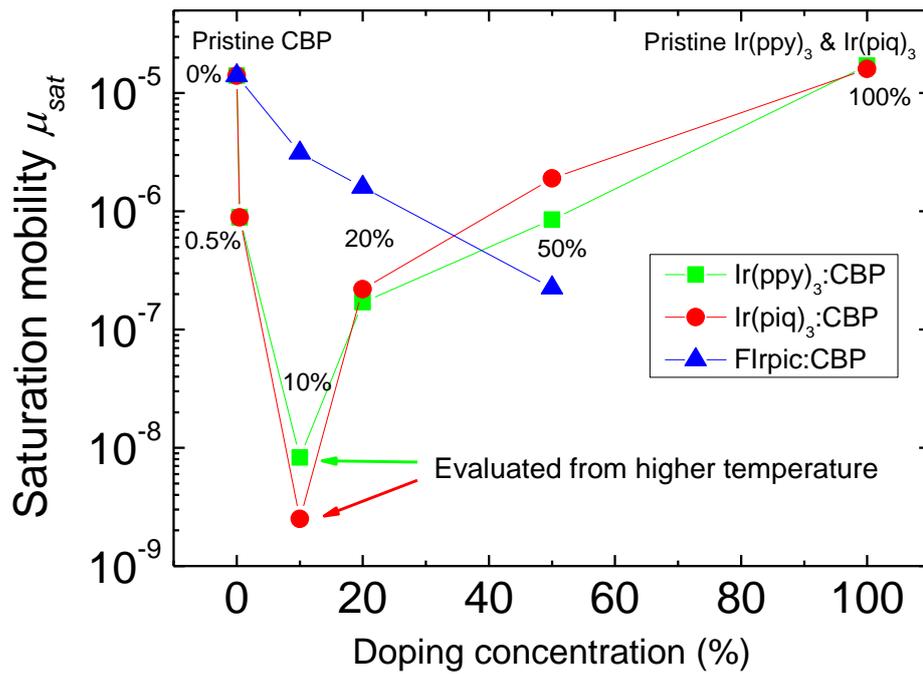
Fig. 5. The saturation hole mobilities of doped CBP films using Ir(ppy)$_3$, Ir(piq)$_3$ and FIrpic as dopants. The doping concentration varies from 0% (undoped CBP) to 100% (neat film of dopant). The data for CBP:Ir(ppy)$_3$ and CBP:Ir(piq)$_3$ were obtained at 390 K.
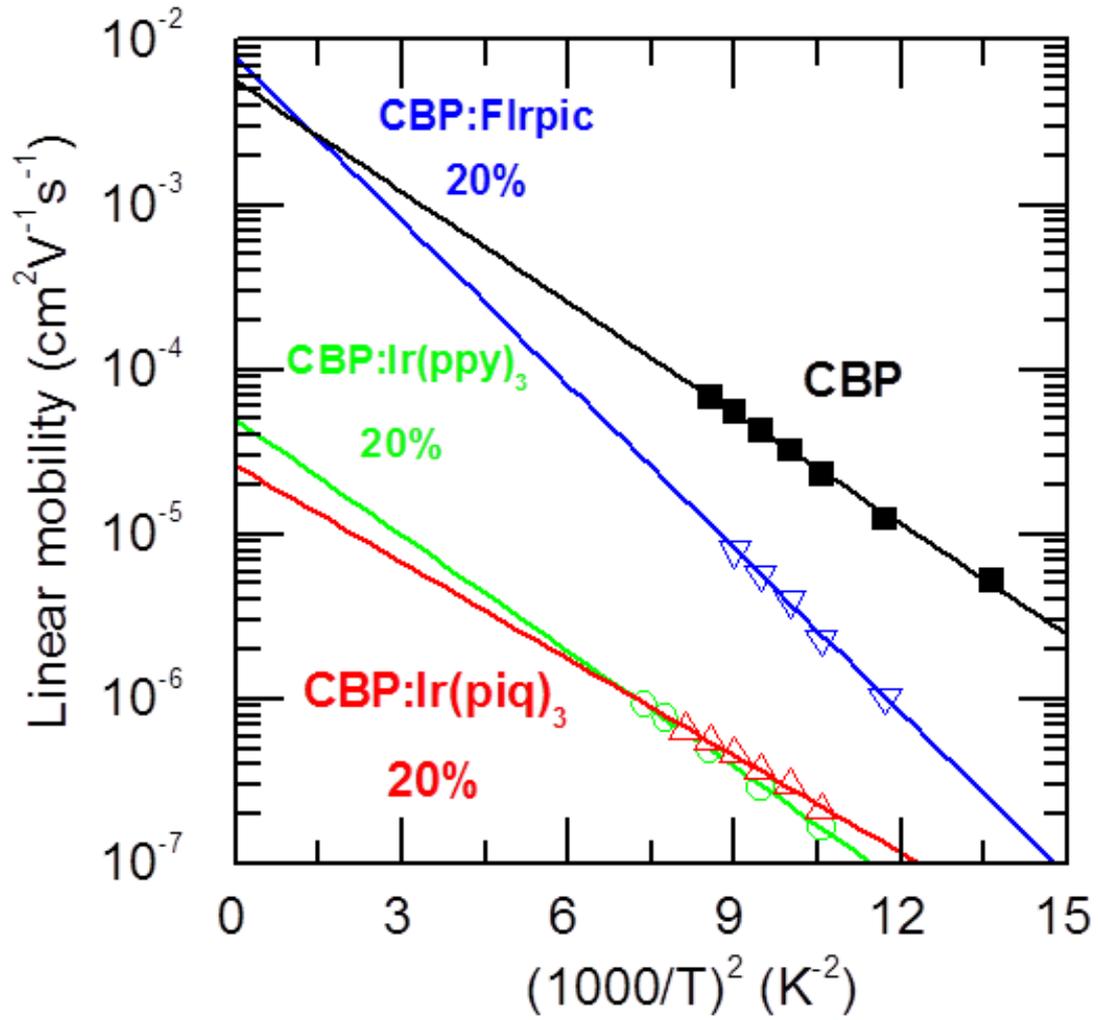
Fig. 6. Plot of μ_{LIN} vs $(1000/T)^2$ for neat CBP and 20% doped CBP films. The slopes and y-intercepts were extracted to obtain transport parameters, which are summarized in Table 2.

	Ir(ppy)$_3$	Ir(piq)$_3$	Ir(Czpy)$_3$	Ir(Czpy)$_2$ (acac)	Ir(Tpa-py)$_3$	Firpic	CBP
μ_{Sat} (cm^{2}V^{-1}s^{-1})	1.7x10^{-5}	1.7x10^{-5}	1.8x10^{-5}	5.5x10^{-6}	1.7x10^{-5}	*	1.8x10^{-5}
HOMO (eV)	5.4 [6]	5.1 [6]	4.9 [15]	5.0 [22]	5.0 [17]	5.8 [6]	6.0 [7]

Table. 1. Hole mobilities of neat films of Ir compounds and CBP as evaluated by TFT technique. The HOMO levels of all compounds are also shown [6-7, 15, 17, 22].

	CBP	CBP:Ir(ppy)$_3$	CBP:Ir(piq)$_3$
μ_∞ (cm^{2}V^{-1}s^{-1})	6x10^{-3}	5x10^{-5}	3x10^{-5}
a (nm)	0.9	1.7	1.8

Table. 2.Transport parameters of CBP and doped CBP estimated from Fig. 6 and Eq.(4).