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Improved Light Emission of Polyfluorene-Type Polymer Light-Emitting Diodes by Thermal Printing and Solution Process

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The emission properties of polyfluorene-type polymer light-emitting diode (PLEDs), using blue emissive poly(9,9-dioctylfluorene) (PFO) and yellow-green emissive poly[9,9-dioctylfluoreny1-2,7-diyl)-co-1,4-benzo-{2,1'-3}thiadiazole)] (F8BT) fabricated by the spin-coating method, the toluene vapor method and the thermal printing method, Poly(2,7-(9,9-di-n-octylfluorene)-alt-(1,4-phenylene-((4-sec-butylphenyl)imino)-1,4-phenylene)) investigated. were (TFB) is useful for buffer layer and a dopant. By using TFB as interlayer of PLED, TFB acts as exciton-blocking layer, thus prevents luminescence quenching. By using TFB with 2-(4-biphenylyl)-5-phenyl-1,3,4-oxadiazole (PBD) as dopants of PFO, current efficiency was improved, compared to PFO only device. This result derives from these materials working as hole and electron transporting molecules. The devices with red emissive Ir complex and near-infrared Pt complex doped in PFO showed red and near-infrared phosphorescent emission, respectively. Current efficiencies of PLEDs with the β phase of PFO fabricated by the thermal printing method and the toluene vapor method were found to have better emission efficiency than that with the amorphous phase of PFO by the spin-coating method. The EL spectra of PLEDs using PFO and PFO:F8BT fabricated by the thermal printing method were polarized. It is expected to improve the characteristics of PLEDs by the optimization of the thermal printing method. We demonstrated improved light emission of PLEDs with the high-quality β phase by the thermal printing method.

Keywords: poly(9,9-dioctylfluorene), β phase, thermal printing method, polymer light-emitting diode, gel

溶液プロセス及び熱転写法にて作製されたポリフルオレン系高分子EL素子 の発光特性改善

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ー般的なスピンコート法で作製したアモルファス相薄膜と、トルエン雰囲気中に曝露する方法や加熱基板に 熱転写する方法などの成膜法を用いて作製した青色発光 poly(9,9-dioctylfluorene) (PFO) や黄緑発光 poly[9,9dioctylfluorenyl-2,7-diyl)-co-1,4-benzo-{2,1'-3}- thiadiazole)] (F8BT)を用いた高分子有機EL素子の発光特性に関 して検討を行った。PFO 薄膜にはクロロホルム溶液に溶解した PFO をスピンコート法により成膜したアモル ファス相薄膜、そのアモルファス相をトルエン雰囲気中に曝露してβ 相に変化させたβ相薄膜、PFO を 1,2,4-トリクロロベンゼン溶液に溶解することで作製したゲル状 PFO を加熱基板上に熱転写する方法で作製したβ 相薄膜を用い、それぞれの高分子 EL 素子の発光効率を検討した結果、β 相薄膜の高分子 EL 素子はアモルフ ァス相薄膜のそれに比べ、発光効率の向上が観測された。三重項発光材料の Ir 錯体及び Pt 錯体をドープした PFO 薄膜を用いた高分子EL素子から、赤色及び近赤外燐光発光が得られ、可視及び近赤外発光高分子EL素 子の作製に成功した。更に、熱転写法から作製した PFO 及び PFO:F8BT 薄膜を用いた高分子EL素子から偏光 発光が得られた。

キーワード:ポリアルキルフルオレン、β相、熱転写法、高分子EL素子、ゲル

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1. Introduction

Organic devices utilizing conducting polymers have attracted considerable interest because of their advantages in large-area device fabrication. Fluorene-type polymer has emerged as an important class of conducting polymers due to their efficient emission and high stabilities [1]. Fluorene-type polymer also has the potential to be in full color emission via energy transfer to longer wavelength emitters in blends with other fluorescent and phosphorescent dyes. Generally, solution process such as the spin-coating method, ink-jet printing [2] is used for forming a thin film of conducting polymer by selecting solvent in which polymer is hardly formed into gel. The characteristics of poly(9,9-dioctylfluorene) [1,3] (PFO) as conducting polymer which has a blue emission and exhibits various morphological behaviors [4-8] have been reported. The characteristics of polymer devices fabricated by solution process are strongly dependent on the self-organized structure as the polymer solution passes from the liquid phase to the solid phase. On the other hand, by dissolving to solvent such as 1,2,4-trichlorobenzene with high boiling point, PFO loses their fluidity gradually and is formed into gel formation. The PFO films and polymer light emitting diodes (PLEDs) using PFO gel by the thermal printing method have been reported [9]. A polymer gel is an intermediate phase between a polymer solution and a solid-state phase.

In this study, we studied the characteristics of blue, yellow-green, red and near-infrared emissive PLEDs using fluorene derivatives as host material. We also demonstrated improved light emission of PLEDs using PFO film with the β phase by the thermal printing method and the toluene vapor method [10], compared to PFO film with the amorphous phase by the spin-coating method.

2. Experimental

Figure 1 shows the typical device structure of PLED and molecular structures. Poly(9,9-dioctyl fluorenyl-2,7-diyl) end capped with dimethylphenyl (PFO, ADS129BE) and poly[9,9-dioctylfluorenyl -2,7-diyl)-co-1,4-benzo-{2,1'-3}-thiadiazole)] (F8BT, ADS133YE) purchased from American Dye Source Inc. were used as emissive layers in this study. A red emissive phosphorescence material and near-infrared emissive phosphorescence material bis[1-(9,9-dimethyl-9H-fluoren -2-yl)-isoquinoline](acetylacetonate)iridium(III) [Ir(fliq)₂ acac] and Pt-tetraphenyl tetrabenzo porphyrin [Pt(tpbp)] was used as a doped dye [11, 12]. The doping concentrations of Ir(fliq)2acac and Pt(tpbp) were 3 and 2 wt% relative to the host material. The substrate was degreased with solvents and cleaned in a UV ozone chamber. First, a poly(ethylene dioxythiophene) poly(styrene sulfuric acid) (PEDOT: PSS) hole injection layer was spin-coated on the indium tin oxide (ITO)coated glass substrate at 45 nm thickness and baked in air at 120°C for 10min.Poly(2,7-(9,9-di-n-octylfluorene)-alt-(1,4-phenylene-((4-sec-butylphenyl)imino)-1,4-phenylene))(TFB,ADS259BE)purchased from American Dye Source Inc. was spin-coated under 10nm thickness as buffer layer and baked at 130°C for 10 min in air and 180°C for 60min in nitrogen gas atmosphere to prevent oxidation. F8BT and PFO layer were spin-coated at 75nm and 100nm thicknesses from chloroform solution and baked at 100 °C for 10min in air.





- Fig. 1. Device structure of PLEDs and molecular structures of PFO, F8BT, TFB, Ir(fliq)₂acac and Pt(tpbp).
- 図 1 高分子EL素子の素子構造と PFO, F8BT, TFB, Ir(fliq)₂acac, Pt(tpbp)の分子構造

Gels using PFO were formed in 1,2,4-trichlorobenzene. Two types of emissive layers were fabricated by the thermal printing method using PFO gel and F8BT doped

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PFO gel as shown in Fig. 2. The doping concentration of F8BT was 5 wt.% relative to PFO. The thickness can be controlled by transfer speed from 20µm/s to 2000µm/s. The substrate temperature and transfer speed were 75.5 °C and 20µm/s, respectively. The solvent was removed by baking the samples in air at 80°C for 20min. The typical thicknesses of emissive layers were 160 - 200nm. For comparison, PFO and F8BT doped PFO layers were fabricated by spin-coating method from chloroform solution. The typical thicknesses of these layers were 120nm. For studying β phase characteristics, we used the toluene vapor method to spin-coated PFO for 20 hours. The cathode consisted of CsF(3nm)/Mg:Ag/Ag was deposited in vacuum at a chamber base pressure of about 10⁻⁴ Pa. Finally, the device was covered with a glass plate and encapsulated by epoxy resin in an argon gas atmosphere to prevent oxidation of the cathode and the organic layer. An active device area of 4 mm² was obtained using a shadow mask.

The EL spectra were measured using a photonic multichannel spectral analyzer (Hamamatsu Photonics PMA-11). The current density-voltage- luminance (I-V-L) characteristics were obtained using a digital multimeter (Keithley 2000), a regulated DC power supply (Kenwood PW36-1.5AD), and a luminance meter (Minolta LS-100). All of the measurements were carried out at room temperature.



Fig. 2. Fabrication process by the thermal printing method using PFOgel.

図2 PFO ゲルを用いた熱転写法による薄膜作製法

3. Result and discussion

We have studied hole and electron transporting effects using TFB and 2-(4-biphenylyl)-5-phenyl-1,3,4-

oxadiazole (PBD). TFB was used as exciton-blocking layer to prevent luminescence quenching [13]. TFB and PBD were doped in PFO for improving property of PLEDs. The β phase effects of PFO have been also investigated. When we use the thermal printing or the toluene vapor method, PFO is formed into β phase [9-10]. In this section, we demonstrate emission characteristics of various PLEDs with fluorene derivatives as host material.

3.1. Characteristics of PLEDs using fluorene derivatives with amorphous phase

At first, we investigated the effect of TFB using as buffer layer. The current density dependence of current efficiency of PLEDs using F8BT as emission layer is shown in Fig. 3. The yellow-green PLEDs using F8BT and F8BT with TFB showed maximum luminances of approximately 850 and 2500 cd/m², respectively.



Fig. 3. Current density dependence of current efficiency of PLEDs using F8BT, F8BT using TFB as buffer layer.

図3 F8BT を用いた高分子 EL 素子の電流密度一効率 特性



- Fig. 4. Current density dependence of current efficiency of PLEDs using PFO, PFO using TFB as buffer layer, PFO:TFB:PBD and PFO:Ir(fliq)₂acac.
- 図4 PFO を用いた高分子EL素子の電流密度一効率 特性



Fig. 5. EL spectra of PLEDs using PFO, F8BT, PFO: Ir(fliq)₂acac and PFO:Pt(tpbp).

図 5 PFO, F8BT, PFO: Ir(fliq)₂acac と PFO:Pt(tpbp)を用い た高分子EL素子の EL スペクトル

At the current density of 100mA/cm², the current efficiencies of approximately 0.6 and 1cd/A were achieved, respectively. This result indicated that TFB worked well as hole transporting layer and exciton-blocking layer. It have been reported that TFB can be used as hole transporting layer instead of PEDOT:PSS [14]. We fabricated PFO:TFB:PBD device with 10:1:1 wt% for improving hole and electron transporting. The current density dependence of current efficiency of PLEDs using various type of PFO and PFO:fluorene derivatives as emission layer is presented in fig. 4. The maximum luminance of PLEDs using PFO, PFO with TFB and PFO:TFB:PBD were approximately 1800, 900 and 2400 cd/m², respectively. At the current density of 100mA/cm², the current efficiencies of approximately 0.15, 0.20 and 1.1cd/A were achieved, respectively. The remarkably improving of the current efficiency of PFO:TFB:PBD device means that TFB and PBD worked as hole and electron transporting layer, respectively.

Figure 5 indicates the visible and near-infrared emissive PLEDs using fluorene derivatives. Fluorene-type polymer also has the potential to exhibit full color emission via energy transfer to longer wavelength emitters in blends with other fluorescent and phosphorescent dyes. In order to utilize the triplet energy transfer from the host to phosphorescent dopants, the high triplet energy of the host is necessary to restrict the backward energy transfer from

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> dopants to host. PFO can be expected to be applied to a host material for red emissive phosphorescent dyes because the band gap energy from singlet ground state (S_0) to lowest exited triplet state (T_1) of PFO is 2.1 eV [15].For PLED with Ir(fliq)2acac doped in PFO, the peak wavelength of approximately 660 nm with a slight shoulder and the red light emission from Ir(fliq)2acac was observed. The red-emissive PLED showed a maximum luminance and a maximum efficiency of more than 1,000 cd/m² and 1 cd/A, respectively (Fig. 4). A gradual decrease of the efficiency was observed at the high current density, which is probably due to triplet-triplet (T-T) annihilation. Near-infrared PLED based on Pt(tpbp) exhibited the EL peak at approximately 770 nm. EL peak for near-infrared PLED is related with triplet energy level of Pt(tpbp) of 1.6 eV.

3.2. Characteristics of PLEDs using polyfluorene with β phase

We reported the possibility of forming films with the β phase by the thermal printing method [9]. In this section, we demonstrated the difference of characteristics of PLEDs fabricated by the thermal printing, the toluene vapor and the spin-coating methods. Figure 6 indicated polarized EL spectra of PLEDs fabricated by the thermal printing method using PFO gel and that doped with F8BT. The spectra were shown for light emitted perpendicular (\perp) and parallel (||) to the transfer direction. The EL intensity perpendicular to the transfer direction I(\perp) was lower than that parallel to the transfer direction I(||). The integrated intensity ratio of I(\perp)/I(||) was estimated to be 0.85. It is noted that this method using polymer gel should be applied to polarized PLEDs.

Current density dependence of current efficiency of PLEDs by the spin-coating, the toluene vapor and the thermal printing methods are presented in Fig. 7. The blue emissive PLEDs with PFO films fabricated by the spin-coating, the toluene vapor and the thermal printing methods showed maximum luminances of approximately 700, 2200 and 2000 cd/m^2 , respectively. For PFO devices, the 421nm peak from the amorphous phase shown in the spin-coated device shifted to the 442nm in the thermal printed device.

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Fig. 6. Polarized EL spectra of PLEDs using PFO gel and PFO:F8BT gel fabricated by the thermal printing method. These devices did not use TFB and PBD.

図6 熱転写法で作製した PFO 及び PFO:F8BT 高分子
 EL素子の偏光ELスペクトル

The yellow-green emissive PLEDs with F8BT-doped PFO films fabricated by spin-coating and thermal printing methods showed maximum luminances of approximately 14,000 and 7,200 cd/m², respectively. It is noted that the luminance from F8BT is dominant, compared to the luminance from PFO in F8BT doped device. In this case, exciton transfer occurred from PFO to F8BT via Förster transfer [16]. In addition, for F8BT-doped PFO device by thermal printing method, the emission from PFO at the wavelength around 450 nm was slightly observed. It is considered that the energy transfer of β phase of PFO to F8BT is not efficient. At the current density of 100mA/cm², the current efficiencies of 0.77 and 0.16cd/A for the PFO devices, and 1.9, 1.7 cd/A for the F8BT doped PFO devices fabricated by the thermal printing and the spin-coating methods were achieved, respectively. For a PFO device fabricated by the toluene vapor method, the current efficiency is almost the same at that by the thermal printing method. PFO with the β phase is expected to get good efficiency. It is noted that the efficiency increased from 0.16 to 0.77 or 0.99cd/A when PFO has the β phase. The β phase and the amorphous phase may be co-existent in the thermal printed and toluene vapor film. Previously Lu *et al.* reported that the β phase acts as a dopant and can be termed self-dopant [17]. Generated self-dopant in the amorphous host allows an efficient energy transfer and promotes hole mobility and leads to more efficient charge recombination. Therefore, high efficiency results from more efficient charge recombination in the β phase device. In addition, that current efficiency didn't increase so much on the F8BT-doped PFO device. This result indicates that the luminance from F8BT is dominant, compared to the luminance from PFO.



Fig. 7. Current density dependence of current efficiency in PLEDs by various fabrication methods.

図7 様々な方法で作製した高分子EL素子の電流密度 一効率特性

4. Summary

We have studied polyfluorene-type PLEDs fabricated by the spin-coating method, the toluene vapor method and the thermal printing method. In the first section, we demonstrated TFB is useful for buffer layer and a dopant using spin-coating method. When we use TFB as inter layer of PLED, TFB acts as exciton-blocking layer, thus prevents luminescence quenching. When we use TFB with PBD as dopants of PFO, better current efficiency was achieved, compared to PFO only device. This result derives from these materials working as hole and electron transporting molecules. The blue and yellow-green PLEDs fabricated by the spin-coating method showed maximum luminances of 2,300 and 2,500cd/m² and maximum efficiencies of approximately 1.1 and 1cd/A, respectively. PLEDs with Ir(fliq)2acac and Pt(tpbp) as phosphorescent dopants doped in PFO exhibited red and near-infrared emissions. The device with Ir(fliq)2acac doped in PFO showed red-emission and a maximum luminance and a maximum efficiency of more than 1,000 cd/m² and 1 cd/A, respectively. The visible and near-infrared emissive PLEDs using fluorene derivatives demonstrated. In the second were section, we

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demonstrated β phase effects of PFO using the thermal printing method and the toluene vapor method. For using the thermal printing method, a PFO film with the β phase can be formed easily. Compared to spin-coated PLEDs with the amorphous phase of PFO, thermal printed PLEDs with the β phase had good current efficiency. The EL spectra of PLEDs using PFO and PFO:F8BT fabricated by the thermal printing method were polarized. The integrated intensity ratio of I(\perp)/I(||) was estimated to be 0.85. It is expected to improve the characteristics of PLEDs by the optimization of the thermal printing method. We demonstrated improved light emission of PLEDs with the β phase by the thermal printing method.

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Yutaka Ohmori was born in 1949. He graduated in 1972 from Department of Electrical Engineering, Faculty of Engineering, Osaka University, Osaka. Japan, where he received the Doctor of Engineering degree. In 1977, he joined Nippon Telegraph Telephone Public Corporation (now NTT Corporation), where he worked mainly research on optical semiconductor devices. In 2000, he became a professor in Collaborative Research Center for Advanced Science and Technology, Electronic Materials and Systems Engineering, Osaka University, and now he is a professor of Center for Advanced Science and Innovation (CASI), where he worked on optical and electrical devices utilizing organic materials including conducting polymers. Dr. Ohmori is a member of the Institute of Electronics, Information and Communication Engineers (IEICE), the Japan Society of Applied Physics (JSAP), American Physical Society (APS), The International Society for Optical Engineering (SPIE) and the Materials Research Society (MRS).