

Electroluminescent characteristics of spin-assembled multilayer films with confined layer structure

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Abstract

Multilayer films composed of poly(*p*-phenylene vinylene) (PPV) as the semiconducting polymer and poly(methacrylic acid) (PMAA) as the insulating polymer were fabricated by spin-assembly method. These films, comprising a confined layer structure, showed that the water contact angles are periodically and distinctly oscillated when the top surface layer is alternated between PPV precursor and PMAA. The turn-on voltage of the multilayer electroluminescent (EL) devices increased from 2.6 V to 9.8 V as the thickness of the PMAA layer inserted between neighboring PPV layers was increased from 0 (i.e., PPV single layer film) to 2.0 nm. Furthermore, the emission peaks in the photoluminescent and EL spectra of these devices were strongly blue-shifted due to excitons formed at the confined PPV layers. Particularly when inserting about 1.0 nm thick PMAA layers, which possibly induced a tunneling effect on the charge carriers (i.e., holes and electrons), these multilayer films decreased the mobility of the hole carriers in the PPV layers with strong hole transporting characteristics, and therefore increased the recombination probability in the emitting layer with confined geometry. As a result, the device efficiency was significantly improved in comparison with that of a PPV single layer device without PMAA layer and with that of devices with relatively thick PMAA layers of 1.4 or 2.0 nm.

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1. Introduction

Recently, there has been considerable interest in the *Layer-by-Layer assembly* method based on dipping process (i.e., dip-assembly method) because of its ability to control the film thickness at a molecular level with the facile insertion of various materials [1–5]. An important advantage of this method is that it enables the preparation of films with controlled thickness, composition and functionality on substrates of different size and shape [1–5]. Additionally, various organic components can be inserted within dip-assembled films through complementary

interactions (i.e., electrostatic, hydrogen-bonding or covalent interaction). Therefore, these films formed have been widely used for polymer electroluminescent (EL) devices [6–9], selective area patterning [10,11], membranes [12,13], photonic crystals [14], and for the surface modification of colloidal particles [15,16]. In particular, the EL devices prepared from dip-assembled multilayers have attracted much attention for the design of devices with improved quantum efficiency. Previous investigation was shown that EL efficiency significantly increases due to the balanced recombination of holes and electrons when hole transporting multilayers composed of poly(*p*-phenylene vinylene) (PPV) and poly(sodium 4-styrenesulfonate) (PSS) are inserted between the indium-tin-oxide (ITO) electrode and spin-coated emitting layer [17]. Furthermore, it was reported that the multiple quantum well (MQW) structured films showing a quantum confinement effect can be prepared

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when the emitting layer is alternately assembled from different energy band-gap materials such as semiconducting polymers and insulating polymers [18,19]. For example, Ohmori et al. reported that the emitting layers with MQW structure using organic molecular-beam deposition could improve the device efficiency by confining the excitons (i.e., electron-hole pairs) in the recombination region and also be reducing the leakage current [20,21]. They also suggested that this structure can induce the blue shift of photoluminescent (PL) and EL spectra due to the quantum confinement effect. However, it is well known that the dip-assembled multilayers have the diffuse layer interface between the adjacent layers due to the high level of polymer interdigitation [22–25], and resultantly this interdiffused layer structure has a difficulty in precisely investigating the quantum confinement effect in the EL devices. On the other hand, the spin-coating assisted method (i.e., spin-assembly method) employing the mechanical force operating onto horizontal direction and the viscous force by water evaporation, as well as the complementary interactions, provides the polymer layers with an extremely smooth surface and dense surface coverage [26,27]. Accordingly, this method notably decreases the polymer chain interdigitation and thus improves the internal structure [28]. On this basis, the spin-assembly method has the advantage of fabricating multilayered EL devices with highly ordered laminated structure similar to MQW structure, and furthermore, of providing a better understanding of the EL properties from quantum confinement effects.

Herein, we report the electro-optical properties of multilayer films with a highly confined layer structure fabricated using the spin-assembly method. For the preparation of these multilayers, PPV as an emitting layer and poly(methacrylic acid) (PMAA) as an insulating layer were assembled using electrostatic interaction. The thickness of the PMAA layer was increased from 0 to 2 nm at a fixed PPV layer thickness of 1.5 ± 0.1 nm. First, we investigated the formation of stratified (or confined) layer structure using surface wettability measurements of the films and then examined the relationship between insulating layer thickness for this structure and EL properties. Based on this investigation, we controlled the insulating layer thickness within multiple laminated structure and obtained a significant improvement in device efficiency.

2. Experimental section

2.1. Materials

Poly(*p*-xylene tetrahydrothiophenium chloride) as a PPV precursor and PMAA ($M_w=15,000$) were purchased from Aldrich and Polysciences, respectively. These polymers were used without any further purification. The concentration of PPV precursor solution used was 10 mg/mL at pH 3.2 to obtain a PPV layer in about 1.5 nm thickness and the solution concentration of PMAA was adjusted to 0.5 mg/mL (for 1.0 nm thickness of PMAA layer), 1 mg/mL (for 1.4 nm) and 2 mg/mL (for 2.0 nm) at pH 3.8, respectively. Ultrapure water (18 M Ω cm) was used for all experiments and the pH of the deposition solutions was adjusted by 1 M HCl or NaOH.

2.2. Buildup of PPV single layer films and multilayer thin films based on PPV

Negatively charged ITO substrates for the deposition of positively charged PPV precursor were initially cleaned by ultrasonification in isopropyl alcohol for 1 hour, followed by immersion in H₂O/H₂O₂/NH₃ (5/1/1) at 80 °C for 1 h, and subsequently dried by N₂ gas purging. For the spin-assembled multilayer films, positively charged PPV precursor solution was completely wetted on the substrates. These procedures can produce a uniform film on a large area except the edge of a substrate. After the solution deposition, the substrate was rotated with a spinner at a fixed rotating speed (typically, 4000 rpm) for about 20 s when sufficient dry of the film was obtained and the substrates were then thoroughly rinsed twice at the speed of 4000 rpm with a plenty of deionized water [26,27]. Negatively charged PMAA layer was also sequentially deposited onto the substrate using the same procedure as mentioned above and all steps were repeated until the desired number of bilayers was obtained. Thirty bilayered PPV/PMAA multilayer films were selected for further measurements of PL and EL spectra as well as current–voltage–light (*I–V–L*) characteristics. For the preparation of PPV single layer, the solvent of PPV precursor was exchanged from water to methanol by using dialysis membrane (MWCO=1000). Single PPV film was fabricated by spin-casting using high concentrated PPV precursor solution. After preparation of multilayered films or single PPV layered films, all samples were annealed at 80 °C for 24 h and then thermally converted from PPV precursor to a conjugated form of PPV at 235 °C for 2 h under high vacuum (1.33×10^{-2} – 10^{-3} Pa). For the preparation of EL devices, aluminum (Al) electrodes with a thickness of about 1500 Å were deposited onto the films through evaporation. The chemical structures of polymers used in this study and a schematic representation of the multilayered EL device are shown in Fig. 1.

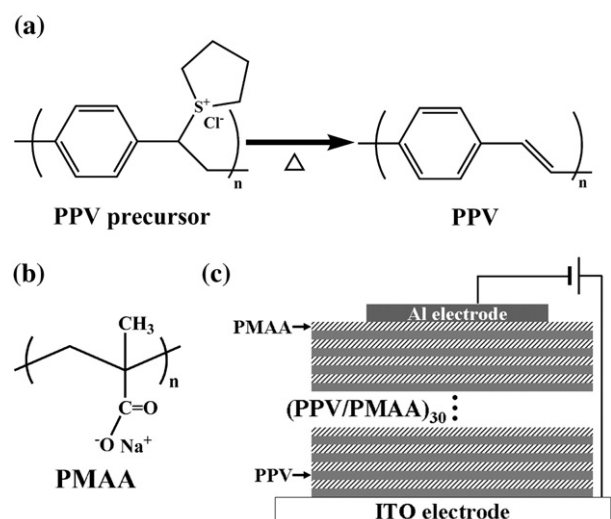


Fig. 1. Chemical structure of the PPV precursor and PMAA and a schematic depicting multilayered EL device.

2.3. Measurement of current–voltage–light characteristics

The I – V – L characteristics were recorded by Keithley 236 Source/Measure Unit for the V – I relationship and by a Newport SIS-SL photodiode for the V – L intensity correlation. Photoluminescence spectra of the films were measured from Shimadzu RF-5000 spectrofluorophotometer by exciting them with the 410 nm line from a 150 W xenon lamp.

2.4. Measurement of contact angle

The water contact angle on the multilayer films was measured using DSA 10-MK2 (KRüss GmbH, Germany) in order to investigate the relationship between internal structure and surface wettability of the multilayer films. In this case, the contact angles were measured after deposition of each layer and subsequent spin washing with deionized water.

2.5. Measurement of film thickness

Silicon wafers were used to measure the total thickness and respective layer thickness of the spin-assembled multilayer films using ellipsometry (L2W15S830, Gaertner Scientific Corp.) with a 632.8 nm He–Ne laser-light. The thickness of PPV precursor ($n=1.56$) or PMAA layer ($n=1.47$) was determined by averaging measurements at more than 10 different points and the thickness of PPV layer ($n=2.10$) was calculated from the thickness change before and after thermal conversion.

3. Results and discussion

3.1. Contact angle measurement

We first investigated the surface quality of spin-assembled multilayers using surface wettability measurement, which is known to be quite sensitive to the chemical and physical properties of the top surface, within the range of a few angstroms (Å) [29–32]. It was previously reported that the

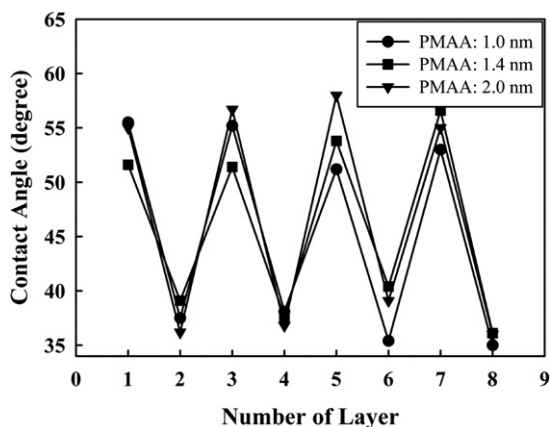


Fig. 2. Water contact angles measured from spin-assembled (PPV/PMAA)_n multilayers. Odd and even numbers indicate the top layers deposited with PPV and PMAA, respectively.

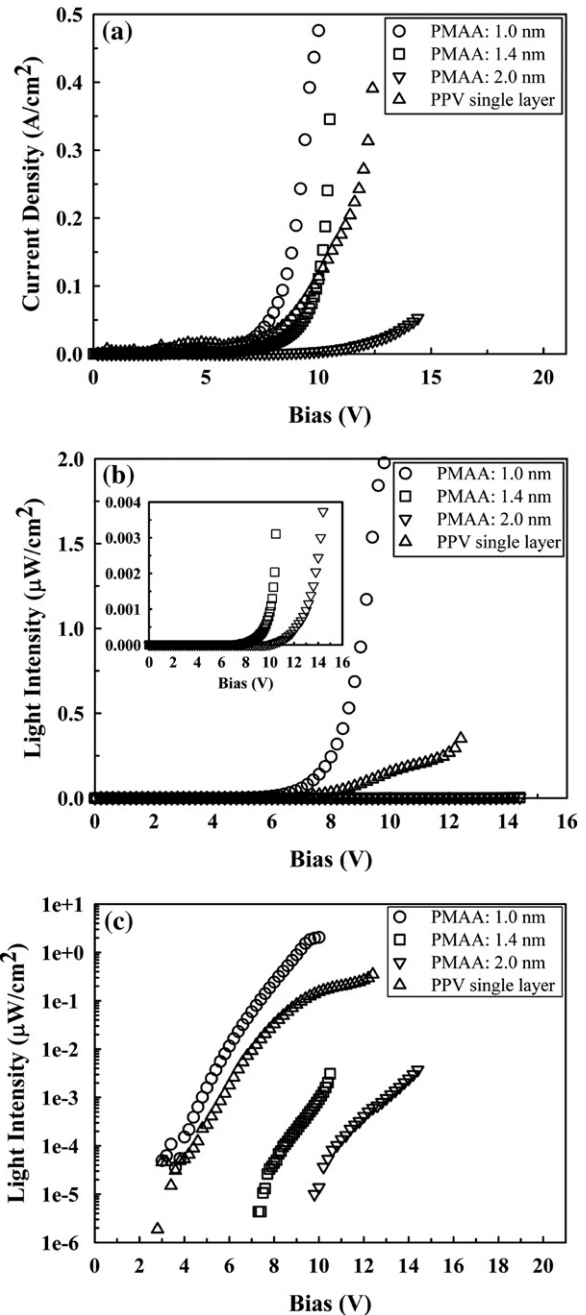


Fig. 3. (a) I – V , (b) L – V and (c) $\log L$ – V plots of ITO/(PPV/PMAA)₃₀/Al devices as a function of thickness of inserted PMAA layers and ITO/PPV/Al device. The inset of (b) shows the extended graph for multilayered devices with PMAA layers of 1.4 and 2.0 nm thicknesses.

water contact angles have an evident oscillation when the top surface polyelectrolyte (PE) layer is alternated between cationic and anionic PE layers using the spin-assembly method [28]. These results mean that the top surface PE layers can effectively screen the chain interdigitation with sublayers and consequently can establish a highly ordered internal structure.

Fig. 2 shows the periodic change in water contact angle of the spin-assembled (PPV/PMAA)_n films when the top surface layer alternated between PPV precursor and PMAA. Odd and

even numbers represent the top surface layers of PPV precursor and PMAA, respectively. In this case, the periodic oscillation of contact angles was evidently observed in the multilayer films with different PMAA layer thicknesses (i.e., 1.0, 1.4 and 2.0 nm). This oscillation behavior in contact angles, as described above, reflects that the spin-assembled (PPV/PMAA)_n films retain a highly ordered or confined layer structure with low degree of interdigitation between the PPV and PMAA layers and that the chemical and physical properties of the top layer therefore almost fully screen those of the sublayers.

3.2. Current-light characteristics

Fig. 3(a) and (b) show the current–voltage and the light–voltage curves of ITO/(PPV/PMAA)₃₀/Al EL devices with increasing the thickness of insulating PMAA layer and ITO/PPV/Al device. The spin-assembled multilayer films with a PMAA layer thickness of 1.0, 1.4 and 2.0 nm, measured by ellipsometry, are denoted as ML10, ML14 and ML20, respectively (Table 1). The increase of PMAA layer thickness from 1.0 nm to 2.0 nm significantly reduced both the light intensity and the current density of multilayered EL devices at the same operating voltage. In this case, the turn-on voltages, at which light began to be emitted, obtained from the $\log L$ – V relationships were 3.0 V for ML10, 7.3 V for ML14 and 9.8 V for ML20 as shown in Fig. 3(c). These voltages were higher than the 2.6 V measured from PPV single layer device without an insulating layer (i.e., PMAA). This increased turn-on voltage strongly implies that the PMAA layers inserted between the respective PPV layers make a significant effect on the mobility of the charge (i.e., hole) carriers. Although the charge carriers injected from the ITO electrode can easily move through a thin PMAA layer of about 1.0 nm by tunneling process, the probability for tunneling decreases exponentially with increasing insulating layer thickness and therefore a higher voltage is required for the tunneling of thicker insulating layers.

3.3. Photoluminescence and electroluminescence spectra

In order to further investigate the electro-optical properties in a confined multilayer structure, we measured the PL and EL spectra of spin-assembled multilayer films under a nitrogen gas atmosphere. Fig. 4(a) shows the change in PL spectra of multilayer films with increasing PMAA layer thickness from 0 (i.e., PPV single layer, SL) to 2.0 nm. The emission peaks of the SL spectrum occurred at about 512 nm and 548 nm, while those of the multilayer films were centered at about 496 nm and

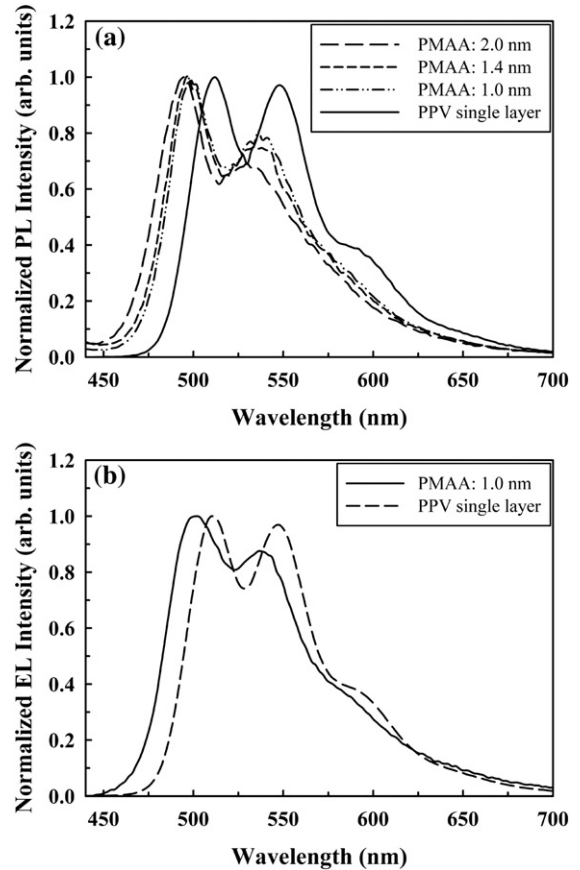


Fig. 4. (a) Photoluminescence spectra of multilayered EL devices with PMAA layers of three different thicknesses and PPV single layered device. (b) Electroluminescence spectra of the (PPV/PMAA)₃₀ device with PMAA layers of 1.0 nm thickness and the PPV single layered device.

538 nm. That is, the peaks obtained from multilayers were slightly shifted to higher energies with increasing PMAA layer thickness. These phenomena were also observed in the EL spectra of ML10 and SL (Fig. 4(b)). Although the EL spectra of ML14 and ML20 were not measured due to the extremely low level of light intensity, the blue-shifted emission peaks of ML10 (502 and 536 nm), in comparison with those of SL (510, 548 nm), support our postulation that the confined structure in the multilayer films can make a significant effect on the exciton energy formed in the PPV layer. More specifically, the PL and EL spectra of films with an organic quantum well structure are blue-shifted by the increased exciton binding energy due to the quantum confinement effect in a potential well [18–21,33,34]. Hong et al. reported that, in the case of adsorbing the relatively thick insulating layer (about 3–5 nm), PL spectra of dip-assembled (PPV/PSS)_n or (PPV/PAA)_n multilayer films are blue-shifted because these insulating layers effectively screen the interpenetration between neighboring PPV layers [35,36]. In this view, it should be noted that the respective PMAA layers of about 1.0 nm thickness can separate PPV layers inducing the blue shift in the PL and EL spectra of spin-assembled EL devices due to the relatively low level of interpenetration between the PPV and PMAA layers (i.e., the distinct interface between the two layers).

Table 1
Thickness of (PPV/PMAA)₃₀ multilayered and PPV single layered EL devices

	PPV single layer(SL)	ML10	ML14	ML20
Total thickness (nm)	75.0±3.0	76.1±1.5	91.2±2.0	107±2.0
Thickness of PPV layer (nm)	75.0±3.0	1.5±0.2	1.6±0.3	1.5±0.1
Thickness of PMAA layer (nm)	0	1.0±0.1	1.4±0.2	2.0±0.2

3.4. Relative quantum efficiency

Fig. 5 shows the relative quantum efficiency of EL devices prepared from (PPV/PMAA)₃₀ multilayer (ML10) and PPV single layer (SL). The total thicknesses of the ML10 and SL devices were 75±3 and 76±1 nm, respectively. Considering that the relative quantum efficiency is given by the slope of the $L-I$ curve (i.e., the ratio of the number of emitted photons and injected electrons), it is noteworthy that the device efficiency of ML10 is about four times higher than that of SL, although the total thickness of the 30-layered PPV inserted within ML10 is only 45 nm due to the inserted PMAA layers (see Table 1). This difference in device efficiency between ML10 and SL can be explained by the confined layer for recombination between the holes and electrons.

Fig. 6 presents a schematic energy diagram for multilayered and single layered EL devices. In this case, the relatively high device efficiency shown in ML10 is caused by the improved recombination probability of the holes and electrons. As already mentioned above, the quantum efficiency of PPV-based EL devices is determined by the efficiency of electron injection at the interface between the emitting layer and Al cathode because PPV has strong hole transporting characteristics [37]. Therefore, the confined PMAA layer in (PPV/PMAA)₃₀ multilayered devices can significantly reduce the mobility of hole carriers due to the high energy barrier of the insulating PMAA layer, as well as the low degree of interdigitation between PPV and PMAA layer. This reduced mobility improves the recombination rate between holes and electrons [17,34,38]. Additionally, the PMAA layers inserted at the interface of PPV/PPV and the PPV/Al electrode can also enhance the quantum efficiency by influencing the energy barrier of the emitting layer. It was reported that the insulator inserted between the emitting layer and Al electrode causes the potential drop and induces a lowering of the electron injection barrier of the emitting layer due to the accumulation of hole carriers at the insulating layer near the Al electrode [39]. Therefore, as shown in Fig. 6(a), it is reasonable to postulate that the confined PPV layers of the spin-assembled EL device can enhance the recombination probability of the holes and electrons in emitting layers due to the charge

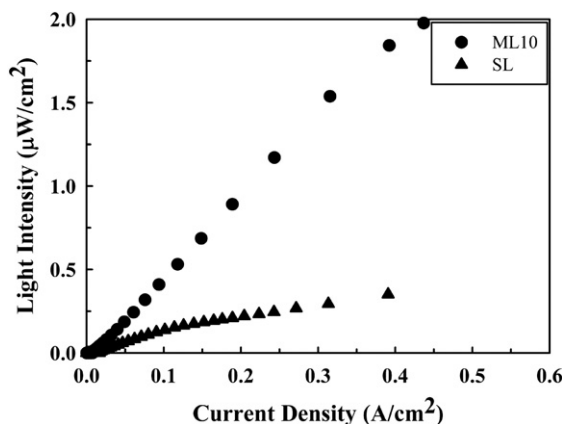


Fig. 5. Relative quantum efficiency of ML10 and SL devices. In this case, ML10 and SL indicate the ITO/(PPV/PMAA)₃₀/Al and ITO/PPV/Al devices, respectively.

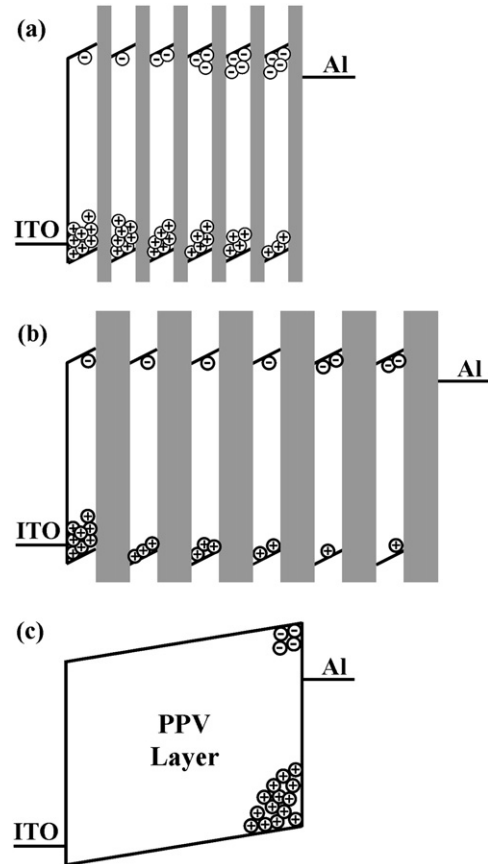


Fig. 6. A schematic energy diagram of (a) ML10, (b) ML20 and (c) SL devices.

accumulation at PPV/PMAA interface by decreasing leakage of hole carriers and increasing injection of electron carriers. However, with the relatively thick insulating layers of ML20, the mobility of the carriers (holes and electrons) injected from the electrodes and the recombination probability are both too low for the light emissions to be detectable (Fig. 6(b)). We also believe that the efficiency of our EL devices will strongly depend on the formation of excitons in the recombination region. If almost all the excitons are formed near the Al cathode by the relatively fast hole mobility, then non-radiative exciton decay significantly increases due to the formation of many defects such as covalent bonding or metal ion diffusion near the Al electrode (Fig. 6(c)) [40]. In this regard, the PMAA insulating layer can control the recombination zone, although not near the electrode, in a multilayered structure by reducing the hole mobility and suppressing the non-radiative decay of excitons via blocking of the direct contact with the Al electrode.

4. Conclusion

We demonstrated that PPV/PMAA multilayers prepared by the spin-assembly method contain an ordered laminated structure with a low degree of interdigitation between adjacent layers and additionally, that these multilayered devices clearly show EL characteristics in association with the quantum confinement effect. First, the presence of a laminated layer structure was confirmed by the periodic and evident oscillations of water

contact angles when the top layer was alternated between PPV and PMAA. Based on this internal structure of the spin-assembled multilayers, increasing the PMAA layer thickness from 1.0 to 2.0 nm increased the turn-on voltage of these devices from 3.0 to 9.8 V, strongly suggesting that the hole injection from the ITO electrode into the multilayers and the hole mobility within the multilayer films can both be controlled by varying the thickness of the inserted PMAA layer. PL and EL spectra of the multilayered devices were also blue-shifted as a result of the quantum confinement effect. Furthermore, the multilayered device with an approximately 1.0 nm thick PMAA layer, which was considered to induce the tunneling effect of the charge carriers (i.e., holes and electrons), significantly improved the relative quantum efficiency in comparison with that of the PPV single layer device without a PMAA layer and with that of devices with relatively thick PMAA layers (1.4 or 2.0 nm). Considering that the electrical properties of EL devices can be easily controlled by varying the thickness of the inserted insulating layer, we believe that spin-assembled multilayer films will enable the possibility for fabrication of highly efficient EL devices.

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