

Control of the Surface Composition of a Conducting-Polymer Complex Film to Tune the Work Function**

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Control of surface composition of a hole-injecting conducting polymer complex, poly(3,4-ethylenedioxy thiophene) (PEDOT) doped with a polystyrene sulfonate (PSS) has been conducted in the spin-cast films. We found that the work function of the polymeric complex films formed via single spin-coating can be greatly increased up to 5.44 eV by increasing the surface concentration of the PSS dopant. As a result, we improved the device efficiency and the lifetime of green emitting polymer light-emitting diodes (PLEDs). This implies that the PSS surface layer of the films spin-cast from the conducting polymer complexes plays a key role in making high performance PLEDs.

1. Introduction

Polymer light-emitting diodes (PLEDs) can be one of the most desirable solutions for large area, high resolution, fast response, and full-color flexible displays based on low-cost solution process, which are currently difficult to achieve by using other types such as liquid crystal or electrophoretic displays.^[1] Tremendous efforts and a great deal of progress have been made to improve the device efficiency and the lifetime in PLEDs. However, it is still very difficult to achieve the high device efficiency and the long lifetime which meet the requirements for commercialization of full-color flat-panel displays.^[2]

Molecular interface engineering between an anode surface and the overlying organic layer can be one of the promising solutions to tune the electronic properties and to prevent the adverse effects of the anode on the device stability. Therefore, it is very important to get some insights to control the hole injection from the anode in the devices. Until now, molecular interface engineering between the anode and the emitting layer (EML) has been done by chemical modification of the anode^[3–5] or introducing a polymeric hole-injection layer (HIL)^[6–10]. Hole-injecting conducting polymer films such as polyaniline and poly(3,4-ethylenedioxy thiophene) (PEDOT) doped with a

polymeric sulfonic acid on indium tin oxide (ITO) layer has been employed because they can improve the hole injection from the ITO (work function: ~ 4.7 – 5.0 eV) and planarize the ITO surface.^[9] A generally used polymeric HIL is composed of PEDOT and polystyrene sulfonate (PSS), called as PEDOT:PSS, which is commercially available from H. C. Starck GmbH.^[11] The PSS acts as a charge compensating counter-ion and stabilizes the *p*-doped PEDOT.^[11,12] PEDOT:PSS forms colloidal particles when it is dispersed in water or alcohol and become processible by simple spin-coating.^[11]

Although PEDOT:PSS films used in PLEDs have a little higher work functions (~ 5.0 – 5.2 eV)^[6,7,11] than that of ITO (~ 4.9 eV), the hole injection could be still a limiting factor in device performance in the case the overlying layer (e.g., hole transporting layer or EML) has much lower highest occupied molecular orbital (HOMO) level with respect to the vacuum level. To overcome such a large hole injection barrier between ITO and the HOMO of the EML, it is important to understand how we can control the surface work function of the HIL layer by molecular surface engineering, which is crucial for efficient hole injection. To date, several approaches to tune the work function of PEDOT:PSS such as addition of a fluorinated ionomer,^[6,7] electrochemical (doping/dedoping) treatment,^[13a] post-deposition treatment and doping,^[13b] and layer-by-layer ionic self-assembly.^[6b,14] Here, we report the tuning of the work function of PEDOT:PSS films by controlling the chemical composition of the film surface layer without adding any chemicals and applying extra treatment. We investigated the surface chemical composition for the spin-cast films of HILs with various ratio of PSS to PEDOT by X-ray photoemission spectroscopy (XPS) and ultraviolet photoelectron spectroscopy (UPS). We found out that the work function of the conventional PEDOT:PSS can be tuned by controlling the ratio of PSS to PEDOT at the film surface, which was conducted by varying the concentration and the molecular weight of the polymeric acid (PSS), and the dispersion medium

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(solvent). Although the degree of surface segregation can also be controlled by heat treatment,^[15] we fixed the film baking condition on a hot plate at 200 °C for 10 min for this study. Although the original PEDOT:PSS (Baytron P VP AI4083) shows the work function of 5.20 eV, the optimized composition showed higher work function of 5.44 eV. The work function tends to be strongly dependent on the concentration of PSS at the film surface. We analyzed the work function and the device efficiency with a function of the relative surface concentration of PSS to PEDOT. Our results clearly showed that the work function tuning can be accomplished by molecular composition tuning at the film surface. The operational lifetime of the devices using the newly reformulated HIL was even better than that of the device using a standard PEDOT:PSS. This implies that the surface control of the conducting polymer HILs doped by the polymeric acid is very crucial to achieve a high device performance.

2. Results and Discussion

The PEDOT:PSS material compositions we used are summarized in Table 1. The PS1H6 and PS1H16Et correspond to the conventional commercial products of H. C. Starck GmbH, i.e., Baytron P VP AI4083 and ET V2, respectively. Other PEDOT:PSS materials were synthesized in H. C. Starck, GmbH according to our special orders. PS1H16Et was synthesized in mixture of 85% ethanol and 15% water, while except for this composition, other materials were synthesized in water and then de-ionized to remove metal ions and sulfate. The PSS contained in aqueous PEDOT:PSS dispersions has two different molecular weights (i.e., $M_w = 48,000$ and $490,000$).

We used XPS to find out surface composition of the various PEDOT:PSS films. The compositional morphology of PEDOT:PSS films is known to be different at the surface than it is in the bulk. Spin-cast PEDOT:PSS films show vertical phase segregation which results in a PSS-rich layer.^[7,15,16] Figure 1 shows a schematic illustration of spin-cast PEDOT:PSS films, which have layered structure with a PSS-rich top layer due to vertical segregation. Figure 2 shows S 2p spectra of the film spin cast from standard Baytron P AI4083 (PS1H6), PS1H3L3, and PS1H6L10 aqueous dispersion. The S 2p spectra show two peaks at 164.6 and 169 eV. Each peak consists of doublet S 2p_{1/2}

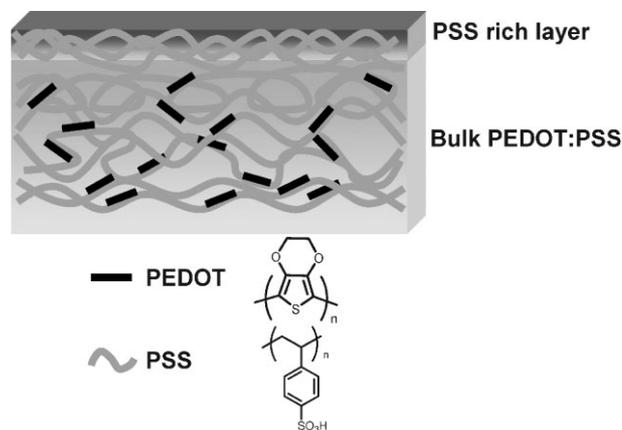


Figure 1. The schematic morphology of spin cast PEDOT:PSS films.

and 2p_{3/2} peaks with an energy separation of 1.2 eV and a fixed area ratio of 0.5. The higher binding energy peak at about 169 eV corresponds to the sulfur atoms in sulfonic acids of PSS. The lower binding energy peak at 164.6 eV corresponds to the sulfur atoms in PEDOT. The asymmetric tail used by Greczynski et al.^[17] for fitting this PEDOT peak was neglected. Instead, a very small peak at about 166.6 eV was added for satisfactory fitting. This peak which can be assigned to an intermediate state^[7] like sulfone ($-\text{SO}_2-$) between atomic-like sulfur (PEDOT) and SO_3^- was neglected in calculating the ratios of PSS to PEDOT at the surface shown in Table 1. The ratios of PSS to PEDOT were calculated using the area ratio of the peaks at 169 eV and 164.6 eV. The information depth can be estimated to about 57 Å from $3\lambda \sin\theta$, where the inelastic mean free path, λ of S 2p electron is 27 Å and take-off angle θ is 45°. Therefore, the ratio of PSS to PEDOT reflects the surface composition within ca. 6 nm. Although we simply assume the PSS-rich layer over the bulk PEDOT-PSS layer^[16] shown in Figure 1, the ratio of PSS to PEDOT at the surface can not be directly converted to the thickness of the PSS-rich layer because PSS exists in both the surface and bulk. J. Hwang et al. estimated that the surface layer thickness of the PEDOT:PSS (1w:4.5w) is around 3.5 nm when the ratio of PEDOT to PSS in the bulk (r) is 0.7, which was obtained from the lightly sputtered PEDOT:PSS using Ar^+ of 250 eV.^[18] However this value deviates so much from the original bulk composition that

Table 1. The PEDOT:PSS compositions and their characteristics of the films and the devices using a green emitting polymer. The ratio of PSS to PEDOT at the surface (PSS/PEDOT@surface), surface work function (WF), film conductivity (S cm^{-1}) by four-point probe conductivity measurement, pH of the dispersion mixtures, and atomic concentration of In (%) at the film surface are presented.

Sample Code	PEDOT	PSS (High MW)	PSS (Low MW)	PSS/PEDOT @surface	WF (eV)	Conductivity (S cm^{-1})	pH	In Conc. (%)
PS1H6	1	6	0	9.44	5.20	6.8×10^{-4}	1.58	0.74
PS1H3L3	1	3	3	14.45	5.29	5.9×10^{-3}	1.59	0.82
PS1L6	1	0	6	14.76	5.32	3.5×10^{-2}	1.60	0.61
PS1H6L6	1	6	6	17.98	5.32	1.0×10^{-4}	1.36	0.55
PS1H6L10	1	6	10	20.93	5.37	4.5×10^{-5}	1.28	0.66
PS1H16Et	1	16	0	48.47	5.44	5.0×10^{-4}	2.11	0.68

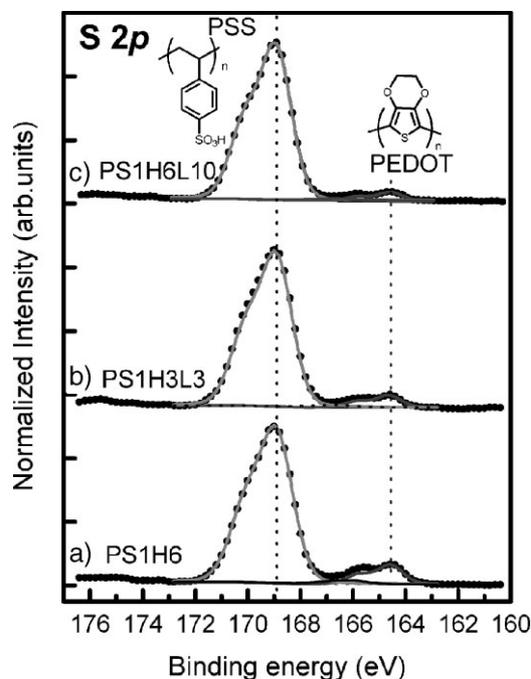


Figure 2. S 2p core level spectra of the PEDOT:PSS with varying the PSS compositions.

the reduction artifact of PSS by sputtering still exist although they used the very low energy Ar^+ .^[18] If we assume that the ratio of PEDOT to PSS in the bulk (r) is close to the ratio of PEDOT to PSS in the solution, the surface layer thickness can be reduced to 2.0 nm using the same equation reported by J. Hwang et al.^[18] By using the same assumption, we also estimated the thickness of the PSS surface layer of our reformulated PEDOT:PSS which ranges from 1.0 to 2.5 nm depending on the compositions.

Figure 3 shows the correlation of the PSS/PEDOT ratio with the work function and the luminous efficiency. First, we observed the effect of the addition of low molecular weight PSS ($M_w = 48,000$) to the PS1H6 (Baytron P VP AI4083). PSS in the conventional PEDOT-PSS dispersions has high molecular weight of $M_w = 490,000$. When low M_w PSS is added, the PSS tends to move upward more easily than high M_w PSS because the low M_w molecules can migrate more easily inside the films compared with the high M_w molecules. As a result, the PSS get richer at the surface as shown in Table 1. As for the PEDOT:PSS mixture prepared in water/ethanol medium (PS1H16Et), the phase segregation of PSS was promoted due to the ethanol even without adding the low M_w PSS. It is interesting to note that the film with higher surface concentration of PSS have higher work function as shown in Figure 3a. Recently, the inverse photoemission spectroscopy combined with UPS showed that the Fermi level of the PEDOT:PSS appears to lie near the middle of the band gap, far from both bands.^[18] The PEDOT:PSS is in a highly p -doped form so that the Fermi level should be closer to the filled states. However, the presence of the insulating PSS at the surface

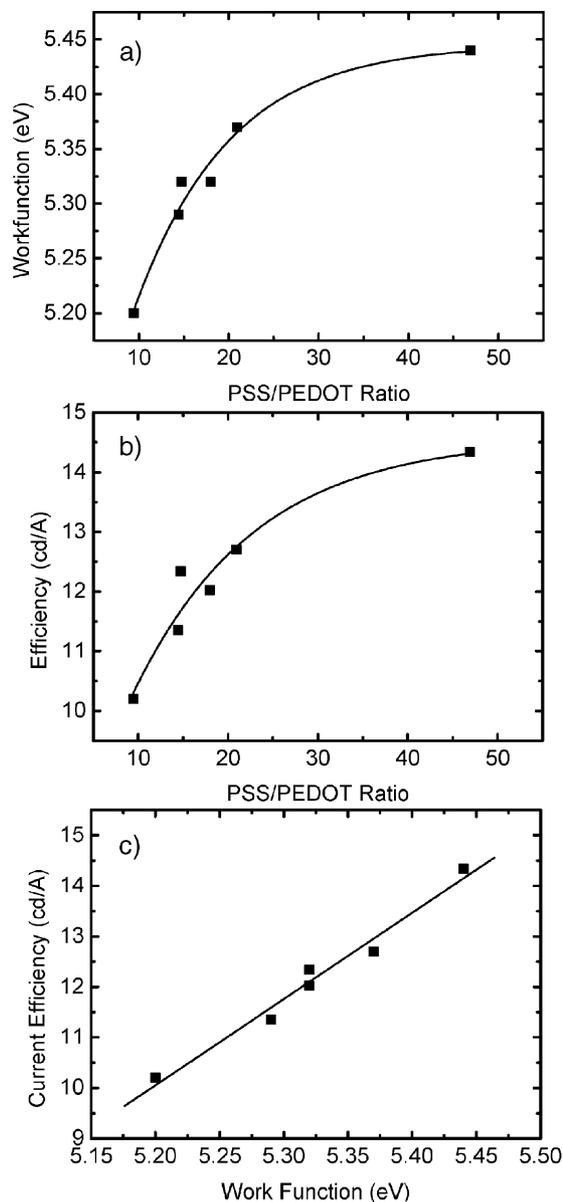


Figure 3. The effect of PSS/PEDOT ratio at the film surface on the film work function and the device efficiency.

strongly attenuates the signal from the bulk PEDOT. Therefore, the removal of the surface layer can lead to a large increase in the density of filled states close to the Fermi level. Greczynski et al. also suggested that the density of state of the doped PEDOT film is suppressed by an overlayer in the surface sensitive measurement.^[16] Enriched PSS layer at the surface can make the surface work function higher than the bulk work function. This implies that the work function of the HIL film can be tuned by the surface layer or especially by the ratio of the polymeric acid (PSS) concentration versus the conducting polymer (PEDOT) concentration at the surface. Figure 3a clearly shows the effect of the surface layer on the work function of the film. We have measured the

conductivity of the spin-cast films by four-point probe conductivity measurement as shown in Table 1. When we compared the PS1H6 with the PS1H6L6 and the PS1H6L10, higher amount of PSS in PEDOT:PSS compositions resulted in lower conductivity. It is noted that when the conductivity of PEDOT:PSS dispersion using a low Mw PSS (PS1L6) is enhanced compared with that of the standard PEDOT:PSS using a high Mw PSS (PS1H6). This finding may imply that low Mw PSS has better chain mobility so that a better connectivity between conducting grains is provided to form better conducting pathways. Although the PS1H16Et film contains the same concentration of PSS with the PS1H6L10 film, the conductivity of PS1H16Et film is higher, which is possibly due to easier formation of conducting grains and their enhanced connectivity by addition of an extra solvent (i.e., ethanol) as reported elsewhere.^[19]

When we fabricated a green emitting PLED using these polymeric complexes (PEDOT:PSS) as HILs and a proprietary green-emitting polyfluorene derivative (HOMO: -5.55 eV, lowest unoccupied molecular orbital: -3.05 eV) as the EML, the device efficiency was also improved as the work function increases (see Fig. 3b). The work function and the current efficiency tend to saturate as the PSS/PEDOT ratio increases. In turn, the device luminous efficiencies were linearly dependant on the work functions of the films as Figure 3c shows. The strong linear relationship between the current efficiency and the work function implies that electrons in the green-emitting devices are more dominated than holes. In fact, the electron mobility of the green-emitting material are higher than hole mobility.^[20] Figure 4a shows the current density versus voltage characteristics of the green-emitting devices using various PEDOT:PSS compositions. Since the major carriers are electrons, the current densities of all the devices above turn-on voltages were quite similar irrespective of the used HILs. However, the device efficiency changes significantly depending on the compositions as shown in Figure 4b. We fabricated hole-only devices using Au top electrode to investigate the effect of the reformulated compositions on the hole injection/transport. Figure 5 shows that the hole current of the devices using reformulated composition was enhanced compared with the standard composition (PS1H6) at forward bias fields. Although the hole currents of the PS1H6L6, PS1H6L10, and PS1H16Et are quite similar, the currents at reverse bias fields shows significant drop with the increasing the PSS content. This is possibly due to charge blocking capability of the insulating PSS. Therefore, in bipolar devices, the surface layer can play an important role in blocking electrons at the PEDOT:PSS/EML interface, resulting in enhanced electron-hole recombination rate. On the other hand, although the work functions of PEDOT:PSS (5.2 – 5.44 eV) are similar to that of Au electrode (5.0 eV), the hole-only device using reformulated compositions of PS1H6 showed high rectification ratios (4.89×10^4 , 6.55×10^4 , 7.25×10^4 at 6V for PS1H6L6, PS1H6L10, and PS1H16Et, respectively).

More interestingly, when we compared the half-lifetime for the devices fabricated using the compositions based on water

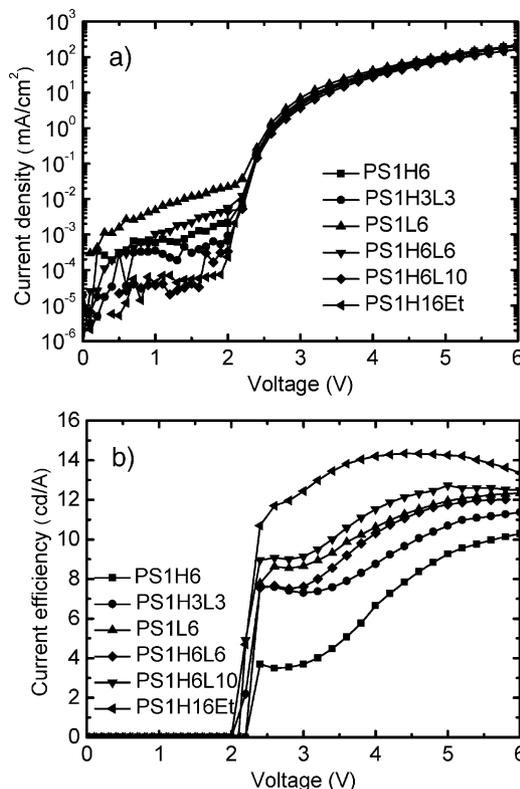


Figure 4. The current and luminescent characteristics of the green-emitting devices with Ba/Al cathodes using various PEDOT:PSS compositions using Ba/Al cathodes. a) The current density versus voltage characteristics; b) The current efficiency versus voltage characteristics.

dispersion, the device lifetime was also improved as the work function increases as Figure 4 shows. These results make it necessary to discuss the role of the PSS surface layer on the device lifetime. Researchers in Cambridge Display Technologies, Ltd at several conferences suggested a degradation mode caused by PEDOT:PSS that the electrons can attack the

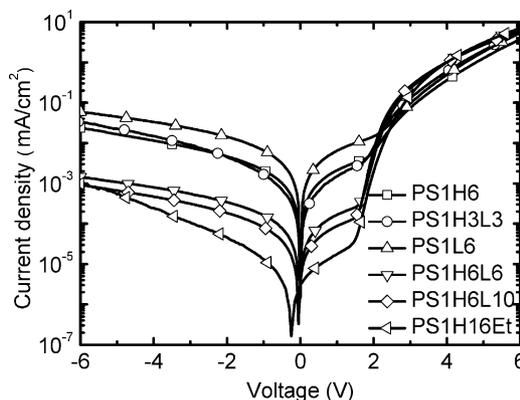


Figure 5. The current density versus voltage characteristics of hole only devices of the green emitting layer with Au top electrodes using various PEDOT:PSS compositions. The periodic diagonal lines stems from the noise during the AFM scanning.

PEDOT:PSS and then residues or by-products (e.g., sulfate released from PSS) diffuse into the light-emitting polymer layer.^[21] Based on the suggested degradation mode, we speculated that the enriched PSS layer might give rise to accelerated degradation of the devices. However, the more PSS concentration in the surface layer at the surface resulted in better device lifetime. On the other hand, we observed that the acidic PSS etches the ITO surface and then In and Sn from the ITO can migrate toward the top surface of the PEDOT:PSS.^[7] Since this can take place in films from all the compositions of PEDOT:PSS, we measured the atomic concentration of In (Table 1) when the sum of atomic C, O, S and In concentrations was set to 100% using XPS. However, the indium surface concentration was less than 1 atom % for all the compositions and was not affected by the PSS concentration in the compositions. We also measured the acidity of the PEDOT:PSS solutions (Table 1). The acidity of the solutions slightly increases as the PSS concentration increases. Although the acidity and the atomic diffusion could be important factors related to the device lifetime, they are not consistent with our device lifetime data. Therefore, we concluded that the enhanced device lifetime is possibly due to an enhanced charge carrier balance by improved hole-injection and electron-blocking capabilities in the PLED devices. Recently, several groups reported use of thin interfacial layers on top of PEDOT:PSS layer which enhances hole injection, electron-blocking, and device lifetime.^[21–23] We speculate that our surface layer may have similar roles with the interfacial layers. The detailed lifetime-enhancing mechanism can be another research topic.

Figure 6 shows the effect of the PSS/PEDOT ratio on the device lifetime. The optimized composition for the device

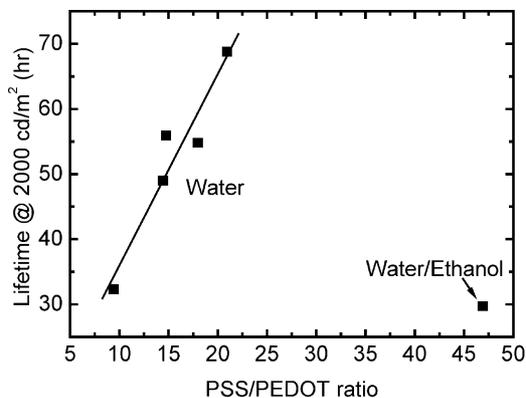


Figure 6. The effect of the PSS/PEDOT ratio on the device lifetime @ 2000 cdm^{-2} .

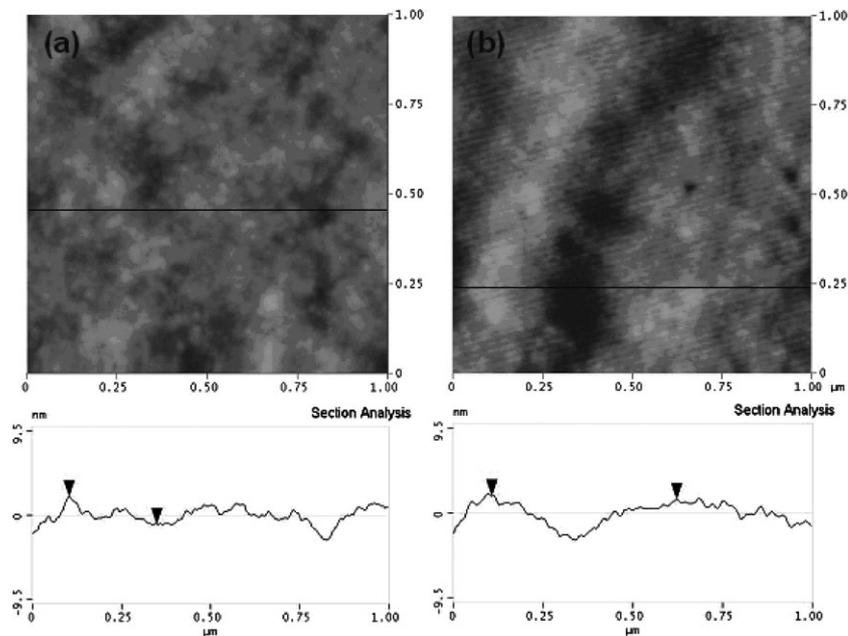


Figure 7. The atomic force microscope (AFM) images of a) PS1H6 and b) PS1H16Et films. The periodic oblique lines of the image (b) stem from the AFM noise during scanning.

lifetime was PS1H6L10 (69 h @ 2000 cdm^{-2}). When we utilized the PS1H6L10 as a HIL, the PS1H6L10 film has the largest surface concentration of PSS (i.e., the ratio of PSS to PEDOT was 20.93) among the compositions based water medium. However, we found out that the film spin-cast from the PEDOT:PSS dispersion in the mixture of ethanol (85%) and water (15%) (PS1H16Et) showed the largest PSS/PEDOT ratio at the surface (48.47). Actually, this composition gave the highest work function (5.44 eV) and the highest device efficiency (14.3 cd A^{-1}). However, the device lifetime was rather reduced compared with the lifetime of the device using the PS1H6L10 as Figure 4 shows. This might be attributed to the large particle size of the PEDOT:PSS in this dispersion. H.C. Starck reported that the PEDOT:PSS synthesized in ethanol/water medium have much larger particle size (>100 nm in solution) compared with that (ca. 55 nm in solution, ca. 25 nm after drying) of conventional PEDOT:PSS.^[11] When we observed the surface morphology by atomic force microscopy (Fig. 7), the film surface of PS1H16Et composition showed very large grooves with long peak-to-peak distance (~500 nm) while the film surface of PS1H6 and other compositions did not show such the long range grooves. This rough surface of the HIL could not provide a planarizing effect on the ITO surface which is crucial for device lifetime.^[9b] This is possibly the reason why the device lifetime was not improved despite the high luminous efficiency.

These results imply that although the control of the interfacial PSS layers formed on the top of the PEDOT:PSS films is very important to tune the work function and thus the device efficiency, the film morphology (e.g., particle size and free volume) could be another important factor to improve the device lifetime.

3. Conclusions

Molecular surface composition engineering of hole-injecting conducting polymer complex films has been conducted by controlling the concentration and molecular weight of a polymeric acid (PSS), and the dispersion medium (solvent). Based on these studies, we tried to get some insights about how we can control the film surface to tune the work function of the spin-cast film. Tuning the work function and thus device performance by single spin-coating of HILs has a great advantage because of the simpler process, compared with the approach using an additional hole-transporting interlayers on top of the HIL.^[18,19] The work function of the conventional PEDOT:PSS can be improved by increasing the surface concentration of PSS on the film surface. This can be achieved by adding extra PSS into the polymeric complexes, by adding low molecular weight PSS, and by changing the dispersion medium (solvent), which resulted in the increase of the work function and thus the device luminous efficiency. In particular, adding low Mw PSS into the PEDOT:PSS compositions was very effective to increase the PSS/PEDOT ratio by promoting the vertical segregation toward the surface. More interestingly, this molecular surface engineering approach resulted in the improvement of device lifetime, which can be attributed to the more balanced electron-hole balance in the device by improving the hole injection and the electron-blocking.

The approach to control the work function of conducting polymer compositions by molecular surface engineering via a single spin-coating should prove to be widely useful in the formation of charge-injecting contact to achieve high performance in other various organic electronic and optoelectronic devices.

4. Experimental

The PEDOT:PSS material compositions we used are summarized in Table 1. The PS1H6 and P1H16Et correspond to the conventional commercial products of H. C. Starck GmbH, i.e., Baytron[®] P VP A14083 and ET V2, respectively. Other PEDOT:PSS materials were synthesized in H. C. Starck, GmbH according to our special orders. PS1H16Et was synthesized in mixture of 85% ethanol and 15% water, while except for this composition, other materials were synthesized in water and then de-ionized to remove metal ions and sulfate. The PSS contained in aqueous PEDOT:PSS dispersions has two different molecular weights (i.e., $M_w = 48,000$ and $490,000$). The PEDOT:PSS dispersions were finally filtered through a $0.45\ \mu\text{m}$ Millex-HV PVDF membrane filter (Millipore, Milford, USA) prior to their use.

The XPS measurements were carried out using a PHI Q2000 system equipped with a monochromatized Al $K\alpha$ source. The work function levels according to the compositions were measured by ultraviolet photoelectron spectroscopy in air (Surface Analyzer, model AC2, Riken Keiki Co., Ltd, Japan). The film thickness for AC2 measurement was ca. 60 nm. The films were baked on a hot plate at $200\ ^\circ\text{C}$ for 10 min and then cooled on the cooling block for 3 min. The samples were measured by AC2 in air within 20 min.

The hole injection layers (HILs) (PEDOT:PSS) were spin-coated to give 60 nm films on top of indium-tin-oxide (ITO)/glass in air and then immediately baked on a hotplate at $200\ ^\circ\text{C}$ for 10 minutes. A

proprietary green-emitting polyfluorene derivative (HOMO: $-5.55\ \text{eV}$, lowest unoccupied molecular orbital: $-3.05\ \text{eV}$) dissolved in toluene was spin-coated on the HIL layers to obtain 80 nm thickness, which was subsequently baked at $100\ ^\circ\text{C}$ for 15 minutes. The 3.5 nm Ba and 150 nm Al were sequentially deposited on the EML under vacuum below 5×10^{-7} Torr. The PLED devices were encapsulated with a glass lid by using a UV curable epoxy resin. The current-voltage-luminescence characteristics were obtained with a Keithley 238 source-measure unit and a Photo Research PR650 spectrophotometer. The device lifetime was recorded by using McScience Polaronix[™] OLED Lifetime Test System. The hole-only devices are fabricated using Au top electrode in the same manner as described above. The current density versus voltage characteristics were measured by a Solartron SI 1287 potentiostat. The measurement sweep was repeatedly done until we get reliable data.

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