Solvation induced morphological effects on the polymer/metal contacts

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Polymer/metal interfaces play an important role in determining the performance of polymer based electronic devices. For most polymer diodes, one of the polymer/metal contacts is usually formed by spin casting a polymer solution onto a metal electrode; we call it polymer-on-metal (POM) contact. The other polymer/metal contact is usually formed by vacuum deposition of metal vapor onto the polymer surface; we call it metal-on-polymer (MOP) contact. In this article, we present evidence that the formation of the POM contact is strongly influenced by the processing of polymer thin films. The POM contact has a greater energy barrier for carrier injection than the MOP contact. The morphology of the polymer thin films, i.e., the relative arrangements of the side groups and conjugated backbone, significantly affects the interfacial characteristics. We also present a simple method of evaluating the morphological effects at the contact. © 2001 American Institute of Physics. [DOI: 10.1063/1.1349859]

I. INTRODUCTION

Poly(p-phenylenevinylene) (PPV) was the first active medium used in polymer electroluminescent devices.¹ However, PPV is infusible and insoluble in common organic solvents. It was processed in the form of soluble monomers and subsequently converted to a polymer active layer. One of the major breakthroughs in polymer light-emitting diode (PLED) research is solution processibility of conjugated polymers, such as poly(2-methoxy-5-(2'-ethyl-hexyloxy)-1, 4-phenylene vinylene (MEH-PPV). MEH-PPV has a similar conjugated backbone as PPV, but it also contains flexible side groups allowing MEH-PPV to be processed by organic solvents, such as xylene.² The easy and low cost solution processibility provides a distinct advantage in using conjugated polymers as electronic materials. Spin coating has become a standard method of producing polymer thin films of excellent quality for different electronic and optoelectronic applications. Since then high performance polymer electronic devices, such as PLED, have attracted attention as low-cost and high-performance devices.

Almost all reported PLEDs have a similar device structure: a polymer thin film sandwiched between two metallic electrodes. Hence, polymer/metal interfaces play an important role in determining the device performance.^{3–5} In the past several years, a number of interdisciplinary studies of polymer surfaces and interfaces have been conducted to a wide variety of π -conjugated polymers and model molecules.^{4–11}

Between two polymer/metal interfaces, the so-called metal-on-polymer (MOP) interface, usually the cathode contact, has been extensively studied.⁴⁻¹⁰ Typically, cathode metals, such as calcium or aluminum, are deposited on top of the polymer film by thermal evaporation under a high

vacuum condition. The metal atoms can readily diffuse into the top surface region of the polymer film and form a diffuse interface with a depth distribution of metal atoms.^{7,8} These metal atoms can sometimes react with the polymer.^{5,7,11} The estimated interfacial region is several nanometers in depth.^{7,8,10}

On the other hand, interfacial formation and properties of the contact between the polymer and the bottom electrode, such as an indium tin oxide (ITO) or ITO/conducting polymer bilayer electrode,^{12,13} have not been carefully examined. Earlier, Abkowitz and his co-workers¹⁴ found that organic/ metal contacts, fabricated by spin coating of organic compound onto Au electrode or by thermal evaporation of Au to the organic film, are different. The top evaporated Au electrodes form injection limited contacts, in contrast with bottom electrodes where the organic is spin coated onto the Au. More recently, Malliaras and co-workers¹⁵ indicated that current–voltage (I-V) curves for forward bias and reverse bias of a gold/polymer/gold hole-only device were not symmetric, which suggests these two contacts are different.

Although most solution processible conjugated polymers are considered as disordered systems, these conjugated polymers still have their own unique morphology, which is strongly influenced by processing conditions. In fact, it has been proved that polymer morphology can be manipulated by the processing conditions, such as using different organic solvents and spin-coating speeds.¹⁶ It was found that during the spin-coating process, relative conformational arrangements of the side groups and conjugated polymer backbone play a very important role in controlling optical and electrical properties of polymer films.^{16,17} Figure 1 shows I-Vcurves and light-voltage (L-V) curves for devices with a similar film thickness but fabricated with different solvents. As can be seen, the onset voltage of current injection (defined as the voltage at which the current "switches on" in a semilog plot) is solvent independent. The device made with tetrahydrofuran (THF) shows a smaller injection current than

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FIG. 1. Current–voltage (I-V) curves and light-voltage (L-V) curves of the devices made with tetrahydrofuran (THF) and dichlorobenzene (DCB), respectively.

the device made with dichlorobenzene (DCB) at the same magnitude of applied voltage. It was also found that the light-emission voltage, V_{L-ON} , (defined as the voltage at which the light switches on in a semilog plot) of the device made with THF (about 1.94 V) is substantially greater than that of the device made with DCB (about 1.75 V).

To gain a better understanding of how the polymer morphology affects the formation of polymer/metal contact, this article presents a simple method in the evaluation of this solvation induced morphological effect on the polymer/metal interface contact. Based on our experimental evidence, we propose an "*effective barrier height*" model to account for the difference in charge injection at the polymer-on-metal (POM) and MOP contacts. We offer a comprehensive discussion for this concept, both qualitatively and quantitatively.

II. EXPERIMENT AND NOTATION

Devices are constructed in a sandwich geometry consisting of a bottom electrode, polymer thin film, and a top electrode. To be consistent with conventional notation, we define the bottom electrode as the anode and the top electrode as the cathode. The direction of bias is also defined as conven-



FIG. 2. Chemical structures of MEH-PPV and different organic solvents.

tional, i.e., forward bias (FB) indicates the bottom electrode is positively biased, while reverse bias (RB) means the top electrode is positively biased.

The metal/polymer/metal device fabrication process consisted of three main steps: the preparation of the bottom electrode, the spin coating of MEH-PPV, and the deposition of the top electrode. The bottom silver or copper electrodes were thermally evaporated onto pre-cleaned glass substrates under a vacuum of $< 10^{-6}$ Torr. The finished glass substrates, coated with the metal strips, were kept immersed in p-xylene until the coating of the polymer to prevent trace contamination.^{15,18} The semiconducting MEH-PPV layer was spin coated on top of the metal strips and subsequently baked at 70 °C for 2 h. Different solvents used were p-xylene, dichlorobenzene, chlorobenzene, cyclohexanone, tetrahydrofuran and chloroform. Figure 2 shows the chemical structure of MEH-PPV and those solvents. Finally, using the same material as that used for the bottom electrode, the top metal electrode was thermally evaporated onto the polymer thin film under a high vacuum. The bottom and top electrode strips ran perpendicular to each other, thereby forming a 4×4 matrix structure. The intersection between two electrode strips formed the active device with an area of about 0.16 mm². Current–voltage (I-V) curves were determined with an HP 4155B semiconductor parameter analyzer with a current resolution of 10 fA.

To further confirm our argument on polymer morphology, reflective absorption Fourier transform infrared (RA-FTIR) spectra were recorded using a Midac M2200 spectrometer equipped with a mercury cadmium telluride detector. P-polarization infrared was employed as the light source for the grazing-angle experiment. In order to reduce the influence from ambient moisture, the instrument as well as the sample compartment was purged by high purity nitrogen gas. Two hundred and fifty-six scans were collected at 4 cm^{-1} resolution for signal averaging. Chrome plated steel plates from Doran Enterprises Inc. were used as substrates for both reference and sample measurements. The substrate was cleaned using a Bunsen burner, and subsequently washed with acetone before the spin-coating process. Polymer films were spin coated from the same polymer solutions as we used to prepare our devices.

III. RESULT AND DISCUSSION

Figure 3 shows I-V curves for Ag/MEH-PPV/Ag devices in which the polymer was processed using different organic solvents. For each device, asymmetric forward bias and reverse bias I-V curves are observed. The injection current for forward bias is (FB) always lower than that for reverse bias (RB). The degree of asymmetry of these I-Vcurves is strongly solvent dependent. Devices processed using aromatic solvents, such as p-xylene, show a smaller degree of asymmetry than those processed using nonaromatic solvents, such as tetrahydrofuran (Table I). For example, the rectification ratio, defined as the ratio of the FB current to the RB current, is about 0.1 for a device processed with p-xylene and 0.02 for a device processed with THF. One exception is the cyclohexanone. Cyclohexanone is a nonaromatic solvent theoretically, however, because of its six carbon-ring structure, it behaves more like an aromatic solvent practically. So in this article cyclohexanone is categorized as an aromatic solvent.

The same phenomena are reproducible using copper as the electrode material (Table I). Figure 4 demonstrates I-V curves for Cu/MEH-PPV/Cu devices.

IV. QUALITATIVE DISCUSSION

Due to their work function, both MEH-PPV/Ag and MEH-PPV/Cu contacts can be considered as Schottky-type



FIG. 3. Current–voltage (I-V) curves for Ag/MEH-PPV/Ag devices using different solvents (a) p-xylene and (b) tetrahydrofuran.

contacts having certain barrier heights.¹⁹ In the past, when estimating the barrier heights at the polymer/metal contacts, the conjugated polymer was treated as an ideal semiconductor with well-defined highest occupied molecular orbital

TABLE I. The estimated values of $\Delta \phi$, extra barrier height component, for MEH-PPV devices processed with different solvents

Devices	Solvent		j_R/j_F	$\Delta \phi \!=\! \phi_F \!-\! \phi_R \; (\text{eV})$
		p-xylene	12±3	0.064 ± 0.006
Ag/MEH-PPV/Ag	Aromatic	Dichlorobenzene	9±2	0.057 ± 0.005
		Chlorobenzene	11 ± 4	0.061 ± 0.009
		Cyclohexanone ^a	9±4	0.057 ± 0.009
	Nonaromatic	Tetrahydrofuran	47±5	0.100 ± 0.003
		chloroform	40 ± 7	0.096 ± 0.004
	Aromatic	p-xylene	9±3	0.057 ± 0.008
Cu/MEH-PPV/Cu				
	Nonaromatic	Tetrahydrofuran	45±5	0.099 ± 0.003

^aCyclohexanone is a nonaromatic solvent theoretically. However, due to its six carbon-ring structure, it behaves more like an aromatic solvent. In this article, cyclohexanone is categorized as an aromatic solvent.

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FIG. 4. Current–voltage (I-V) curves for Cu/MEH-PPV/Cu devices using different solvents (a) p-xylene and (b) tetrahydrofuran.

(HOMO) and lowest unoccupied molecular orbital energies arising from its conjugated electronic structure. Theoretically, the major contribution to the semiconducting characteristics of conjugated polymers is from π -conjugated segments along polymer backbones.⁴ In reality, this ideal case is somewhat naive. Most conjugated polymers, such as MEH-PPV, contain two major structures: π -conjugated backbones and alkyl side groups containing mainly saturated σ bonds (Fig. 2). Therefore, the metal/polymer contacts also consist of two components: contacts with the π -conjugated segments and contacts with nonconjugated, σ -bonded alkyl segments. We now define an "effective barrier height," ϕ , containing these two components: an intrinsic component, ϕ_i , corresponding to, among other things, the direct contacts between metal atoms and the π -conjugated segments, and an extra component, $\Delta \phi$, corresponding to direct contacts between metal atoms and the nonconjugated alkyl segments. Hence $\phi = \phi_i + \Delta \phi$. The "intrinsic" component corresponds to the barrier height which is the "true" energy value



FIG. 5. Schematic illustrations of polymer morphology at polymer-on-metal contacts fabricated with (a) aromatic solvents and (b) nonaromatic solvents.

required to overcome the barrier at the metal/conjugated polymer backbone contact. For a given polymer and metal, it is expected that ϕ_i is a well-defined value at a fixed temperature. On the other hand, the second term $\Delta \phi$ is an extra component which takes into account the contribution due to the physical separation between the metal atoms and the conjugated segments by the bulky side chains at the interface. Unlike the first term, the second one is dependent on detailed molecular conformations and local interfacial morphology. These are very sensitive to the fabrication conditions, such as different types of solvents used to prepare polymer solutions and the different thin-film processing conditions.^{16,17}

We have already reported that solvation-induced morphological effects significantly influence a device's optical and electrical properties.^{16,17} It is found that different solvents preferentially solvate different structures along the polymer chains, i.e., "like dissolve like" from chemistry point of view, thereby forming different molecular conformations in solutions as well as spin-coated thin films. With aromatic solvents, such as p-xylene, the conjugated segments of MEH-PPV are preferentially solvated by the organic solvents, resulting in a polymer conformation which is termed as the "aromatic (AR)" conformation. The dominant AR conformation allows the polymer to have a greater possibility of intimate contacts between the conjugated groups and the metal atoms [Fig. 5(a)] and subsequently facilitates charge injection by minimizing the contribution of the second term in the effective barrier height. On the other hand, by using nonaromatic solvents, such as tetrahydrofuran, solvation effects prefer to orient the nonaromatic side groups outside and "trap" the conjugated groups inside, thus developing a "nonaromatic (NAR)" conformation. As a result, the conjugated backbones are mainly shielded by those bulky alkyl side groups; those bulky side groups prevent intimate contacts between conduction groups in the polymer backbone and the metal atoms [Fig. 5(b)]. A higher effective



FIG. 6. RA-FTIR spectra of MEH-PPV films with AR (dichlorobenzene as solvent) and NAR (tetrahydrofuran as solvent) conformations.

barrier height, ϕ , will be expected as a result of a higher value of $\Delta \phi$.

The changes in polymer morphology caused by the processing conditions are further supported by experimental results obtained from RA-FTIR measurements. RA-FTIR spectra of MEH-PPV films with AR (dichlorobenzene as the solvent) and NAR (tetrahydrofuran as the solvent) conformations are shown in Fig. 6. In the high frequency region the peaks of 2958, 2930, 2872 and 2857 cm^{-1} , correspond to the -CH₃ asymmetric stretching, the -CH₂-asymmetric stretchthe $-CH_3$ symmetric stretching, ing, and the -CH₂-symmetric stretching vibration, respectively. The ratios of the peak intensities of $2958-2930 \text{ cm}^{-1}$, and 2872- 2857 cm^{-1} do show obvious difference, caused by the (average) orientation of the molecular dipole moment. Since the orientation of the dipole moments is related to the packing of alkyl side chains of MEH-PPV molecule, this provides strong evidence that the morphology of polymer films is strongly influenced by the different types of solvents. We are still working on the RA-FTIR experiment and more results will be reported separately.

The solvation-induced morphological effects on the metal/polymer contacts are also dependent on interface formation processes. For the POM interface obtained by spin coating of a polymer solution onto the metallic electrode, we have found that morphological effects have significant effects on interface properties. When a polymer film is being formed, polymer conformations in the solution are most likely to be carried over into the polymer film, especially when those solvents with a low-boiling point, such as tetrahydrofuran and chloroform, are used.^{16,17} In addition, there is little diffusion between the bottom metal layer and the polymer film (spin casting is carried out at room temperature), so the POM contact can be considered as a sharp interface with a distinct metal/polymer boundary.

In contrast, for metal-on-polymer contacts obtained by thermal evaporation of metal onto a polymer thin film, the morphological effects are likely to become less important. The interface formation involves the diffusion of metallic atoms into the polymer surface, and the MOP contact is thus a diffuse-type contact. The nanoscale diffusion profile of metallic atoms is likely to overwhelm the morphological effects discussed earlier. In addition, a chemical reaction may happen at the interface which might play a role in enhancing the interface formation.^{9–11} The barrier height obtained for those MOP contacts should be closer to the true value, i.e., ϕ_i . Based on the difference in the nature of the contacts, the direct comparison of I-V characteristics under RB and FB can give an estimate of the morphology contribution to the barrier height.

V. QUANTITATIVE DISCUSSION

Thermionic emission was identified as the charge injection mechanism of PLEDs by several groups.^{20,21} At zero bias, the density of holes with sufficient energy to overcome the energy barrier at the metal and polymer interface and jump into the π orbital of the polymer is

$$p_{0} = \int_{-\infty}^{E_{\text{HOMO}}} g(\boldsymbol{\epsilon}) [1 - f(\boldsymbol{\epsilon})] d\boldsymbol{\epsilon}$$

$$\propto \int_{-\infty}^{E_{\text{HOMO}}/k_{0}T} \frac{[(E_{\text{HOMO}} - \boldsymbol{\epsilon})/k_{0}T]^{1/2} \cdot d(\boldsymbol{\epsilon}/k_{0}T)}{1 + \exp[(\boldsymbol{\epsilon}_{F} - \boldsymbol{\epsilon})/k_{0}T]}, \quad (1)$$

where E_{HOMO} stands for the energy of the highest occupied molecular orbital of MEH-PPV, ϵ_F is the work function of metals, $f(\epsilon)$ is the Fermi distribution, $g(\epsilon)$ is the density of states, ϵ is the energy, k_o is the Boltzmann constant and T is the absolute temperature. Then p_0 can be expressed as

$$p_0 \propto \exp\left(\frac{E_{\text{HOMO}} - \epsilon_F}{k_0 T}\right) = \exp\left(\frac{-\phi}{k_0 T}\right),$$
 (2)

where $\phi = E_{\text{HOMO}} - \epsilon_F$ is the effective energy barrier for hole injection at the polymer and metal electrode contact. When a voltage *V* is applied, Eq. (2) can be rewritten as

$$p \propto \exp\left(\frac{E_{\text{HOMO}} - \epsilon_F + eV}{k_0 T}\right) = \exp\left(\frac{-\phi + eV}{k_0 T}\right).$$
 (3)

For an individual single-carrier (hole-only) device, it is reasonable to assume that the average velocity of charge carriers under the same magnitude of forward bias and reverse bias should be same. Since j = nev (where *n* is the density of charge carriers, *v* is the velocity of the charge carriers), the difference in the current density *j* under forward and reverse bias should reflect the difference in the density of injected charges. Then we have

$$j \propto p \propto \exp\left(\frac{-\phi + eV}{k_0 T}\right).$$
 (4)

Under bias, we can rewrite Eq. (4) as

$$j_F \propto I_F \propto \exp\left(\frac{-\phi_F + eV}{k_0 T}\right),$$
 (5a)

$$j_R \propto I_R \propto \exp\left(\frac{-\phi_R + eV}{k_0 T}\right),$$
 (5b)

where subscripts *F* and *R* refer to forward and reverse bias, respectively, ϕ_F and ϕ_R refer to the effective barrier height

for forward and reverse bias condition, respectively. Hence, the ratio of current density (j) or current (I) at the same bias is

$$j_{R}/j_{F} = I_{R}/I_{F} = \exp\left(\frac{-\phi_{R} + eV}{k_{0}T}\right) / \exp\left(\frac{-\phi_{F} + eV}{k_{0}T}\right) = \exp\left(\frac{\phi_{F} - \phi_{R}}{k_{0}T}\right).$$
(6)

The difference in the barrier heights at the two contacts can be estimated from the ratio of reverse and forward current under the same magnitude of forward and reverse bias (Table I). If we further assume that $\phi_R \approx \phi_i$, then $\phi_F - \phi_R$ is $\Delta \phi$, the extra contribution to the barrier height due to the morphological effects at the interface. This component is estimated to be higher for devices made with nonaromatic solvents, such as tetrahydrofuran, than those made with aromatic solvents, such as p-xylene by roughly 50%. For a polymer film with AR conformations, $\Delta \phi$ is about 0.06 eV, while for a polymer film with NAR conformations, it is about 0.1 eV.

Recently, some "unexpected" results were reported for hole-only devices with a metal/polymer/metal sandwich structure. For an Au/MEH-PPV/Au device, I-V curves under forward bias and reverse bias were expected to be symmetric, however an asymmetric I-V curve was observed.¹⁵ In addition it was also observed that this asymmetry had a built-in potential on the order of a few tenths of a volt. Meanwhile, for a Cu/ MEH-PPV/Al device, an asymmetric I-V curve was expected because barrier heights at two contacts calculated based on work functions of the metals were different by 0.2 eV (the work function for Cu and Al is 4.5 and 4.3 eV, respectively). However, a nearly symmetric I-Vcurve was reported.²² In both cases, origins of those unexpected results were not well explained.

It is most likely that those results can be well explained if the solvation-induced morphological effects are considered. The POM contact has a stronger morphological component ($\Delta \phi$) than the MOP contact. This extra contribution to the barrier height at the contact between the polymer and the bottom Au electrode may result in the detected built-in potential. In the case of the Cu/MEH-PPV/Al device, which is fabricated with a nonaromatic solvent chloroform, it is very probable that $\Delta \phi$ becomes significant. As a result the actual barrier height at the Cu contact is increased and becomes comparable to that of the Al contact. The I-V curve accidentally becomes symmetric.

VI. CONCLUSION

Solvation-induced morphological effects play an important role in controlling the properties of contacts between MEH-PPV and metallic electrodes. Injection barrier heights are highly sensitive to formation processes of polymer/metal contacts and conformational states of polymer chains. The polymer-on-metal contacts formed by spin-coating process have a greater barrier height than the metal-on-polymer contacts formed by thermal evaporation. The presence of alkyl side groups at a polymer/metal contact results in an extra component to the barrier height. This extra barrier height component changes the efficiency of charge injection and subsequently the overall device performance. An aromatic solvent can significantly reduce this extra barrier height component. Furthermore, our results suggest new directions for the design of dual function polymers having improved solubility while simultaneously preserving the charge injection efficiency.

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