

Novel Light-Emitting Devices Based on Pyridine-Containing Conjugated Polymers

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Abstract— We present novel light-emitting devices based on several pyridine-containing conjugated polymers and copolymers in various device configurations. The high electron affinity of pyridine-based polymers improves stability and electron transport properties of the polymers and enables the use of relatively stable metals such as Al as electron injecting contacts. Bilayer devices utilizing poly(9-vinyl carbazole) (PVK) as a hole-transporting/electron-blocking polymer show dramatically improved efficiency and brightness as compared to single layer devices. This is attributed to charge confinement and exciplex emission at the PVK/emitting polymer interface. The incorporation of conducting polyaniline network electrode into PVK reduces the device turn-on voltage significantly while maintaining the high efficiency. Two novel device configurations that enable the use of high work function metals as electrodes are pointed out.

I. INTRODUCTION

SINCE its discovery in 1990 in poly(*p*-phenylene vinylene) (PPV) [1], conjugated polymer electroluminescence (EL) has been considered an important property with many potential applications. Electroluminescence combined with other unique properties of polymers, such as processibility, bandgap tunability, and mechanical flexibility, make conjugated polymers excellent candidates for low-cost large area display applications. In addition to PPV, a variety of PPV derivatives and other conjugated polymers and copolymers have been found to exhibit electroluminescent properties [2], [3]. Light-emitting devices incorporating these materials have demonstrated all the necessary colors needed for display applications.

Over the past few years, the performance of polymer based light-emitting devices has been improved dramatically. A number of techniques have been developed to improve the quantum efficiency. One technique is to use a low work function metal, such as Ca, as the electron injecting electrode (cathode) [4]. The double charge injection mechanism of polymer light-emitting diodes (LED's) requires the matching of the cathode (anode) work function to the corresponding

LUMO (HOMO) level of the polymer in order to achieve efficient charge injection. The relatively low electron affinity of most conjugated polymers requires metals with very low work functions to achieve efficient electron injection. However, since low work function metals are generally oxygen reactive, devices with a low work function cathode are usually unstable. Thus, polymers with high electron affinities are highly desirable.

Pyridine-based conjugated polymers have been shown to be promising candidates for light-emitting devices [5], [6] due to their high electron affinity. As compared to phenylene-based analogs, the pyridine-based polymers are more resistant to oxidation and show better electron transport properties. Fig. 1 shows the structures of the pyridine-containing polymers and copolymers used in this study, namely poly(*p*-pyridine) (PPy), poly(*p*-pyridyl vinylene) (PPyV), and copolymers of PPyV and PPV [PPyVP(R)₂V] with various functional side groups $R = C_{12}H_{25}$, $OC_{16}H_{33}$, $COOC_{12}H_{25}$. With respect to the π electronic levels, $-C_{12}H_{25}$ is slightly electron donating; $-OC_{16}H_{33}$ is electron donating; and $-COOC_{12}H_{25}$ is electron withdrawing. The pyridine-based polymers are highly luminescent, especially the copolymers. The internal photoluminescence quantum efficiencies of the copolymers have been measured [7] under ambient environment to be 75–90% in solutions and 18–30% in films, with the exception of the $-OC_{16}H_{33}$ copolymer. The electron donating nature of $-OC_{16}H_{33}$ makes the copolymer more susceptible to oxidation. For example, carbonyl formation has been identified in the $-OC_{16}H_{33}$ copolymer films. As a result, the PL quantum efficiency is only 2% in film even though it is high ($\sim 80\%$) in solution. In contrast, the strapped copolymer containing $C_{10}H_{20}$ as a bridging group, Fig. 1(d), does not oxidize as readily. Also the strapped copolymer shows the effects of less aggregation as compared to the “usual” copolymers.

Another common technique to improve device performance is to incorporate charge transporting layers in a multilayer device structure. The charge transporting layer enhances the transport of one type of charge while blocking the other, thus achieving balanced charge injection and transport. In addition, the charge transport layer spatially confines the emission zone away from the electrode, avoiding nonradiative quenching effects near electrodes. To date the highest efficiency polymer light-emitting devices reported are multilayer devices [8].

Here we present light-emitting devices based on the pyridine-containing polymers and copolymers in various

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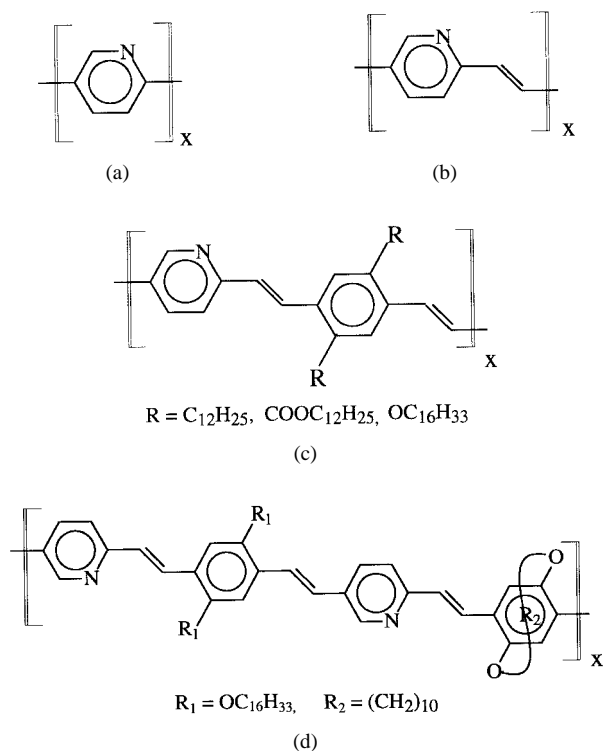


Fig. 1. Structures of pyridine based conjugated polymers and copolymers: (a) poly(*p*-pyridine) (PPy), (b) poly(*p*-pyridyl vinylene) (PPyV), (c) copolymers of PPyV and PPV derivatives [PPyVP(*R*)₂V] with various functional side groups $R = \text{C}_{12}\text{H}_{25}, \text{OC}_{16}\text{H}_{33}, \text{COOC}_{12}\text{H}_{25}$, and (d) strapped copolymer (@PPyVPV).

device configurations. One advantage of the pyridine based polymers is the high electron affinity. For example, the electron affinity of PPy is ~ 3.5 eV [9], which is ~ 0.78 eV higher than its phenylene based analog poly(*p*-phenylene) (PPP) and is ~ 0.9 eV higher than the most widely used PPV [2]. The high electron affinity of pyridine based polymers enables the use of relatively stable conductors such as Al as electron injecting contacts and the development of a number of novel device configurations. Taking advantage of the better electron transport properties of the pyridine-containing polymers, we fabricate bilayer devices utilizing poly(9-vinyl carbazole) (PVK) as a hole transporting/electron blocking layer, which improves the device efficiency and brightness significantly due to the charge confinement and exciplex emission at the PVK/emitting polymer interface. The incorporation of a conducting polyaniline network electrode within PVK reduces the device turn-on voltage significantly while maintaining the high efficiency. The high electron affinity of the pyridine-based polymers enables the use of ITO as an electron injecting contact (instead of an hole injecting contact as in typical polymer light-emitting diodes) to fabricate inverted light-emitting devices [10]. A metal with much higher work function, such as Au, is used as a hole injecting contact. By inserting an "insulating" layer on both side of the emitting polymer, we fabricated symmetrically configured AC light-emitting (SCALE) devices [6] which work under both forward and reverse DC bias as well as in AC modes. Both the inverted and the SCALE devices are capable of eliminating the use of

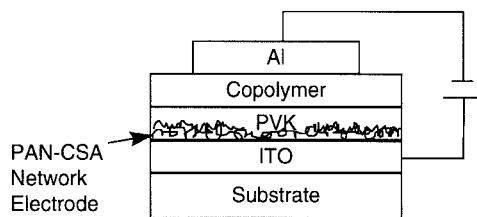


Fig. 2. Schematic structure of a bilayer device with conducting polyaniline network electrode.

low work function metals as electrodes, potentially improving the device operating stability and shelf lifetime.

II. EXPERIMENT

The synthesis of the pyridine-containing polymers has been reported earlier [11]–[13]. For single layer devices, the emitting layer was spin-cast at 1000–2000 r/min from solutions in formic acid (for PPy and PPyV) or xylenes (for copolymers) (with a concentration of ~ 10 mg/ml) onto precleaned patterned ITO substrates with a sheet resistance of $15 \Omega/\text{square}$. For bilayer devices, the PVK layer was spin-coated onto the ITO substrate from solution in tetrahydrofuran (THF) (~ 10 mg/ml) at ~ 3000 r/min. The emitting layer was then spin coated on top of the PVK layer from appropriate solutions. As the solvent for the emitting layer can not dissolve PVK, no significant intermixing of the two polymers is expected. The conducting polyaniline network electrode was formed by spin-casting from a blend (1:4 ratio) of camphor sulfonic acid doped polyaniline (PAN-CSA) and a low molecular weight host polymer poly(methyl methacrylate) (PMMA) (from Aldrich Chemical Company) in *m*-cresol. The host polymer PMMA was subsequently washed away by xylenes. As xylenes is an excellent solvent for PMMA but a poor solvent for PAN-CSA, the extraction of the PMMA left a porous network of PAN-CSA. The PVK and emitting layers were subsequently coated as in the bilayer device. The PVK layer is expected to partially fill the PAN-CSA network, resulting in a structure schematically illustrated in Fig. 2. Similar effects have been found for the formation of PAN-CSA network within a PPV derivative [14]. All the spin coating procedures were carried out inside a class 100 cleanroom. The polymer films were not annealed. The top metal electrode was deposited by vacuum evaporation at a pressure below 10^{-6} torr. To prevent damage to the polymers, the substrate was mounted on a cold-water cooled surface during evaporation.

Absorption spectra were measured on spin-cast films using a Perkin-Elmer Lambda 19 UV/VIS/NIR spectrometer. Photoluminescence (PL) and EL were measured using a PTI fluorometer (model QM-1). The current-voltage (I - V) characteristics were measured simultaneously with EL using two Keithley model 195A multimeters while dc voltage was applied by a HP model 6218A DC power supply. Quantum efficiency and brightness were measured using a calibrated photodiode (UDT UV100). We note that all the device testing procedures were performed in air on as-made devices without any encapsulation. The lifetimes for most devices were modest, approximately 3 h at initial brightness of $\sim 200 \text{ cd/m}^2$. The

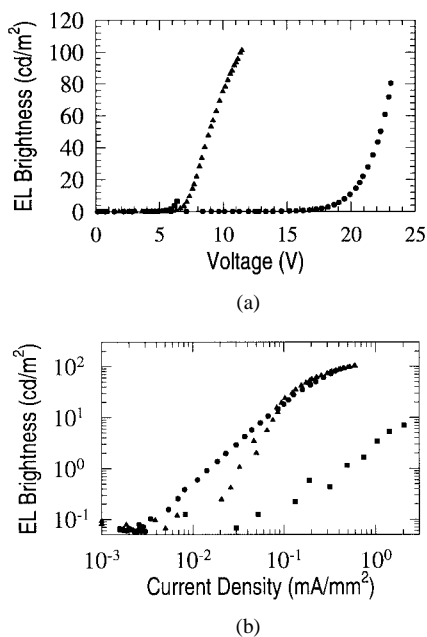


Fig. 3. Comparison of (a) light–voltage and (b) light–current characteristics for a single layer device (square), a bilayer device (circle), and a bilayer device with PAN-CSA network (triangle).

lifetimes were substantially longer when the devices were operated under low power conditions.

III. RESULTS AND DISCUSSION

Fig. 3 compares the electroluminescence–voltage (EL– V) and electroluminescence–current (EL– I) characteristics for a single layer device, a bilayer device, and a bilayer device with PAN-CSA network electrode using the strapped copolymer as emitting layer. As compared to those of the single layer devices, the internal quantum efficiencies of the bilayer device increase more than two orders of magnitude, reaching $\sim 0.3\%$. The brightness of the bilayer device follows closely with the current density (not shown), reaching ~ 50 cd/m² at ~ 0.2 mA/mm² (~ 22 V) and ~ 300 cd/m² at ~ 2 mA/mm² (~ 27 V). Fig. 4 shows an operating seven-bar numeric display using the bilayer structure. PVK is a well known hole transporting/electron blocking polymer. In addition to the effects of enhancing the transport of holes injected from the anode, PVK blocks the transport of electrons injected from the cathode such that the electrons accumulate at the PVK/copolymer interface. This charge confinement greatly enhances the probability of radiative recombination. In addition, the PVK layer separates the recombination zone from the metal electrode so that the radiative recombination is protected against the nonradiative quenching at the metal/polymer interfaces.

A side effect of using the PVK layer is that it increases the device operating voltage substantially. One effective way to reduce the device turn-on voltage is to use a high surface network electrode [14]. The concept behind the network electrode is that a rough electrode will create a nonuniform high electric field that enhances the charge injection. This technique has been successfully applied to PPV based devices [14]. By applying this technique to the PVK layer, the device operating voltage decreased significantly. For the devices shown

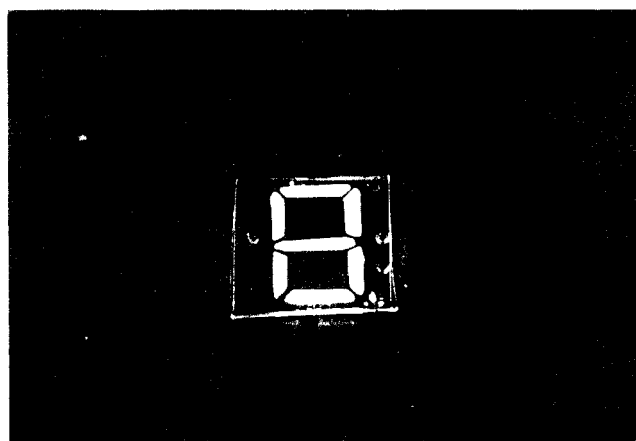


Fig. 4. A photograph of an operating seven-bar numeric display using ITO/PVK/strapped copolymer/Al bilayer structure.

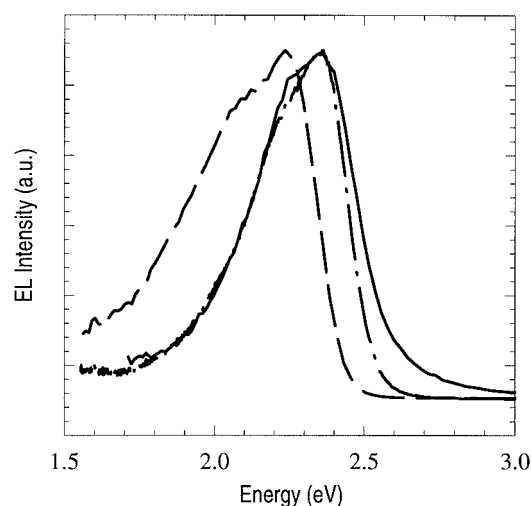


Fig. 5. Normalized EL spectra of the single layer device (dashed line), the bilayer device (solid line), and the bilayer device with network electrode (dash-dotted line).

here, the device operating voltage reduced significantly [see Fig. 3(a)]. The brightness of the bilayer device with network electrode reaches ~ 50 cd/m² at only ~ 9 V, instead of ~ 22 V for bilayer devices without network electrode. Since the incorporation of the PAN-CSA network electrode does not modify the PVK/copolymer interface, the high quantum efficiency and brightness of the bilayer device are maintained [see Fig. 3(b)]. Thus, the incorporation of the network electrode to the bilayer device dramatically improves the power efficiency (light output power/electrical input power) from $\sim 0.04\%$ to $\sim 0.13\%$. The bilayer devices with and without PAN-CSA network electrode show similar EL spectra, which are blue shifted as compared to that of the single layer device, see Fig. 5. The slightly reduced intensity at high energy tail for the device with network electrode is probably due to absorption by the PAN-CSA.

The species that are responsible for the light generation in the bilayer device is attributed to exciplexes formed at the PVK/copolymer interface, and are identified by the PL measurements [17]. Fig. 6 compares the PL of pure copolymer, pure PVK, and bilayers of PVK/copolymer. The PL of PVK film (dotted lines) excited at 3.6 eV shows an emission peak

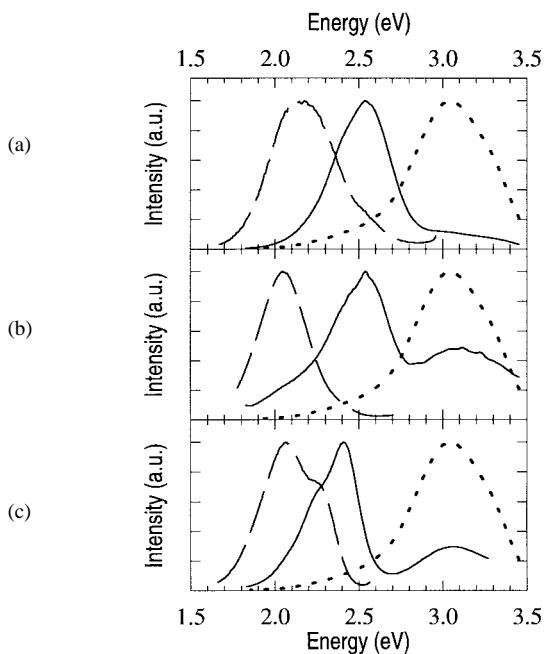


Fig. 6. Photoluminescence of a single layer of PVK (dotted line), a single layer of copolymer (dashed line), and a bilayer of PVK/copolymer (solid line). (a) PPyVP(C₁₂H₂₅)₂V, (b) PPyVP(COOC₁₂H₂₅)₂V, and (c) strapped copolymer.

at 3.06 eV, agrees well with the value reported in the literature [15], [16]. The PL of the copolymers with $R = C_{12}H_{25}$ and COOC₁₂H₂₅ peaks at ~ 2.15 and ~ 2.03 eV, respectively. In contrast, the PL of strapped copolymer film shows a shoulder at 2.25 eV, in addition to the main peak at ~ 2.03 eV. The peak of the film PL is clearly red-shifted as compared to solution PL for all the copolymers, which has been attributed to the aggregate formation in the film [18], [19].

The solid lines in Fig. 6 are the PL spectra for the PVK/copolymer bilayer films excited at 3.6 eV, an energy that is greater than the band gap of PVK. In each case, more prominently in Fig. 6(b) and (c), there is PL emission at the same energy as the PVK PL emission (3.1 eV). However, the main feature in the PL of the bilayer films is located at 2.5 eV for the copolymers with $R = C_{12}H_{25}$ and COOC₁₂H₂₅ and 2.4 eV for the strapped copolymer. Emission at these energies is not observed for individual films of either PVK or the copolymers indicating that the emission is due to a completely different species, and is attributed to the exciplex. When the excitation energy is lowered below 3.4 eV (band gap of PVK) the emission due to the exciplex is drastically reduced. In addition, varying the concentration or thickness of the copolymer or PVK films in the bilayer configuration will change the relative strengths of the exciplex peak and PVK peak.

The assignment of exciplex emission is supported by optical absorption and photoluminescence excitation (PLE) measurements. Fig. 7 shows the optical absorbance for the single and bilayer systems. Each of the plots show the absorbance of a single layer of PVK, a single layer of the copolymer, and the bilayer PVK/copolymer films. The onset of PVK absorption is at 3.5 eV and shows two spectral features at 3.6 and 3.75 eV similar to previous reports [15], [16]. The absorbance of the

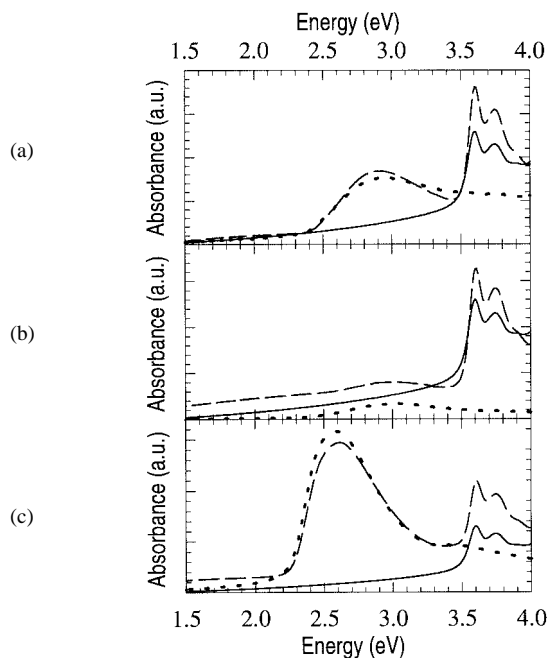


Fig. 7. Absorbance of a single layer of PVK (solid line), a single layer of copolymer (dotted line), and a bilayer of PVK/copolymer (dashed line). (a) PPyVP(COOC₁₂H₂₅)₂V, (b) PPyVP(C₁₂H₂₅)₂V, and (c) strapped copolymer.

bilayer film in each case is the sum of that of each individual component. No new ground to excited state transitions are present. The shape of the photoluminescence excitation (PLE) versus energy curve (not shown) is nearly the same as the absorbance versus energy curve.

We note that a shoulder at 2.2 eV appears in both single and bilayer PL for the strapped copolymer (Fig. 6). Although the strapped and the corresponding unstrapped copolymer show similar features in solution PL, no shoulder was found in the film PL for the unstrapped copolymer. We attribute the shoulder to the partial break-up of the aggregate formation due to the wrap-around of the C₁₀H₂₀ chains. This assignment is supported by the PL measurements of the blends of the strapped copolymer in PMMA. When the concentration of the strapped copolymer decreases, the strength of the shoulder grows and is blue shifted toward the solution PL. It eventually becomes the dominant feature, indicating that the PL is dominated by unaggregated sites in the dilute blend. The same shoulder also appears in the bilayer film of PVK/strapped copolymer indicating that in addition to exciplexes, unaggregated sites of pure strapped copolymer also contribute to the PL of the bilayer film. The EL of the PVK/strapped copolymer bilayer device follows closely with the PL of the bilayer film suggesting that both the exciplex and unaggregated sites of pure strapped copolymer contribute to the light generation unlike the case of other "usual" copolymers [17] in which the exciplex is responsible entirely for EL emission.

The high electron affinity of the pyridine-based polymers enables novel device configurations such as inverted light-emitting devices [10] that are capable of eliminating the use of low work function metals. Poly(*p*-pyridine) (PPy) has an electron affinity of ~ 3.5 eV [9], which allows metals with

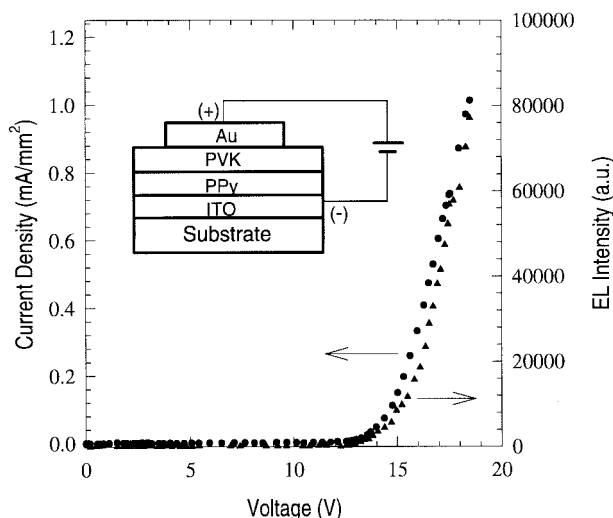


Fig. 8. Current–voltage characteristics of an inverted light-emitting devices with PPy as emitting layer and PVK as hole transporting layer. Inset: schematic device structure of such an inverted device.

relatively high work functions as electron injecting contacts. In the inverted light-emitting devices, ITO was used as an electron injecting contact, unlike most polymer LED's which use ITO as the hole injecting contacts. Au, a metal with even higher work function, was used as a hole injecting electrode. The inverted (–)ITO/PPy/Au(+) devices show improved device performance including quantum efficiency, brightness, operating stability and storage lifetimes as compared to the usual (+)ITO/PPy/Al(–) device. By inserting a PVK layer in between the PPy and Au, the device performance improves further. Fig. 8 shows the I – V characteristics of a typical (–)ITO/PPy/PVK/Au(+) device. The device has a typical turn-on voltage of ~ 10 V, and light follows closely with current after turn on. The device structure is shown schematically in Fig. 8 inset.

Other novel device configurations such as SCALE (symmetrically configured AC light-emitting) devices [6] also allow the elimination of low work function metals as electrodes. The SCALE devices consist of an active emitting layer sandwiched between two “insulating” polymer layers which are able to accept both electrons and holes. This configuration enables the SCALE devices to work under both forward and reverse DC bias. Due to the relatively fast dynamic response, the devices can also be operated in AC modes. This unusual behavior is attributed to the effects of charge accumulation at the emitting polymer/insulating polymer interfaces.

IV. CONCLUSION

In summary, pyridine containing conjugated polymers and copolymers are excellent candidates for polymer light-emitting devices. The high electron affinity of pyridine based polymers enables the use of relatively stable metals such as Al as efficient electron injecting contacts. Taking advantages of the better electron transport properties of the pyridine-containing polymers, we fabricate bilayer devices utilizing PVK as hole transporting/electron blocking polymer. The bilayer device structure improves the device quantum efficiency

and brightness significantly due to the charge confinement and exciplex emission at the PVK/emitting polymer interface. The incorporation of the conducting polyaniline network electrode within PVK reduces the device turn-on voltage significantly while maintaining the high efficiency and brightness of the bilayer device. The high electron affinity of the pyridine-based polymers enables the fabrication of novel devices such as inverted light emitting devices and SCALE devices which are capable of eliminating the use of low work function metals as electrodes, potentially improving the device operating stability and shelf lifetime.

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Dr. MacDiarmid is presently on the editorial boards of *Inorganic Syntheses*, *Synthesis*, and *Reactivity in Inorganic and Metal-Organic Chemistry*, and *New Polymeric Materials*. He is also a Regional Editor of *Synthetic Metals*. In the past, he was the Editor of the series of monographs, *Organometallic Compounds of the Group IV Elements* (New York: Marcel Dekker), and Vol. XVII of *Inorganic Synthesis*. He has also served on the Editorial Board of *Inorganic Chemistry*. During 1975, he served as the Chairman of the Inorganic Division of the American Chemical Society; he has also served several terms as a counselor and as a member of the Board of Directors of the Philadelphia Section of the American Society. He is the recipient of numerous awards and honorary degrees both nationally and internationally.



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