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On-Demand Photochemical Stabilization of Doping in Light-Emitting Electrochemical Cells

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Abstract

A highly functional p-n junction doping structure can be realized within a light-emitting electrochemical cell under applied voltage *via* ion redistribution and electrochemical doping. This doping structure will however dissipate when the formation voltage is removed due to the mobility of the dopant counter-ions. A number of concepts aimed at a spatial immobilization of the ions and the related stabilization of the doping structure have been presented, but they all suffer from long and poorly controlled stabilization periods and/or unpractical operational conditions. Here, we introduce a markedly fast and easy-to-control stabilization procedure involving the inclusion of a UV-sensitive photo-initiator compound into a carefully tuned active material in an light-emitting electrochemical cell device, and demonstrate that it is possible to cross-link the ions and stabilize the p-n junction doping *via* a short UV exposure step executed at room temperature.

1. Introduction

The concept of joining a p-type and n-type doped inorganic semiconductor together to attain a p-n junction has proven to be immensely useful in the field of microelectronics, and it is today utilized in almost all semiconductor device technologies.[1] The p-n junction concept has also been imported into the emerging field of organic electronics, notably in high-performance organic light-emitting diodes (OLEDs), where the doped layers comprise organic semiconductors in the form of small molecules intermixed with dopant molecules.[2-5] The fabrication of the various layers in OLEDs is done by vacuum processing that can be quite cost and energy extensive, but an alternative organic semiconductor – the conjugated polymer – offers the important advantage of solution processing that can be extremely efficient, particularly if executed in a roll-to-roll fashion.[6-11] Unfortunately, it has proven difficult to attain a stable p-n junction in conjugated polymer materials, as the dopant counter-

ions (that stabilize the doping) typically are mobile and tend to diffuse in the soft, disordered and often porous conjugated polymer matrix with a concomitant dissipation of the p-n junction doping structure.[12-14]

The light-emitting electrochemical cell (LEC) comprises a blend of a conjugated polymer and mobile ions as the active material sandwiched between two electrodes.[15-25] An LEC actually makes use of the mobility of the ions for the *in-situ* electrochemical formation of doped conjugated polymer regions at the electrode interfaces, and the subsequent establishment of a light-emitting p-n junction within the bulk of the active material, under the direction of an externally applied voltage.[26,27] However, the p-n junction in LECs is dynamic and only stable as long as the applied voltage remains, and for applications where a fast, repeatable response and rectification of current and light emission are required this represents a problem.

A number of methods aimed at the stabilization of the p-n junction structure in LECs have been demonstrated. Gao and Heeger introduced the so-called “frozen junction” method, which utilizes the temperature dependence of the ionic mobility.[28,29] The p-n junction is built at a temperature at which the ions are mobile, and the device is thereafter cooled to a temperature at which the ions effectively become immobile and the p-n junction as a consequence is stabilized.[30-36] Lonergan and co-workers stabilized a p-n junction at the interface between a cationic and an anionic conjugated polyelectrolyte by solvent-induced removal of the mobile counter-ions,[37] while Bazan *et al.* utilized *in-situ* boron-fluoride chemistry on a similar device structure for the same purpose.[38] Finally, Leger and Bartholomew[39] conceptualized the more generic “chemical stabilization” approach, which utilizes designed dopant counter-ions that can be physically immobilized *via* cross-linking polymerization reactions that presumably are initiated by electrochemical means.[39-43]

Here, we expand on the latter work and design devices in which the doping stabilization can be conveniently executed by a short UV-light exposure. This is accomplished by including a UV-sensitive photo-initiator material into a tuned active material blend comprising an ion-pair monomer and an ion-transport material, both endowed with an acrylic functional group, in addition to the conjugated polymer. We show that such devices can be photochemically stabilized via a sub-minute UV exposure time to exhibit fast turn-on, high current rectification ratio, and good efficiency following days of storage under open-circuit conditions.

2. Experimental

Fig. 1. Top: Chemical structure of the materials in the active layer of the LEC devices under study. Bottom: Schematic presentation of the the UV-induced decomposition of the DMPA photo-initiator, and the initiation, propagation and termination of the polymerization process.

The employed conjugated polymer is a decyloxyphenyl substituted poly(1,4-phenylene vinylene) termed superyellow (SY, Merck, Darmstadt, DE), and it was used as received. The ion-pair monomer 2-(methacryloyloxy)ethyl trimethylammonium 2-acrylamido-2-methyl-1-propane sulfonate (METMA/AMPS) was synthesized in our laboratory by using commercially available starting materials, as detailed in a previous publication.[40] The cross-linkable ion-transport material poly(ethylene glycol) (poly-PEO; $M_w = 575$ g/mol, Aldrich, Steinheim, DE) and the photo-initiator 2,2-dimethoxy-2-phenylacetophenone (DMPA, Aldrich, Steinheim, DE) were used as received. Fig. 1 presents the chemical structure of the compounds employed for the active material in LEC devices in the top part.

Four different master solutions were prepared: 5 mg/ml SY in toluene, 10 mg/ml poly-PEO in cyclohexanone, 2.5 mg/ml METMA/AMPS in cyclohexanone, and 10 mg/ml DMPA in cyclohexanone. An active material solution was prepared by mixing the master solutions in a “dry mass” ratio of SY:poly-PEO:METMA/AMPS:DMPA = 1:0.17:0.06:0.003. The active material solution was stirred on a magnetic hot plate for 5 h at $T = 323$ K immediately before film fabrication. Indium tin oxide (ITO) coated glass substrates (1.5×1.5 cm², 20 ohms/square, Thin Film Devices, Anaheim, CA) were cleaned under ambient conditions by sequential ultrasonic treatments in detergent (Extran MA 01, Merck), acetone, and isopropanol. The active material solution was deposited onto the ITO substrates by spin-coating at 800 rpm for 1 min. The resulting active-material film was thereafter dried on a hot plate at $T = 323$ K for 5 h. The thickness of the active material was 120 nm as established by atomic force microscopy. Four rectangular Al electrodes were deposited through a shadow mask on top of the film by thermal evaporation at $p < 2 \times 10^{-4}$ Pa to finish off the fabrication of the ITO/active-material/Al sandwich cell LECs. The active area of each LEC device was 13 mm².

A system comprising a computer-controlled source-measure unit (Keithley 2400) and a calibrated photodiode with an eye response filter (Hamamatsu Photonics), connected through a current-to-voltage amplifier to a HP 34401A meter, was employed for driving the LEC devices and measuring the resulting optoelectronic response. A commercially available hand-held UV lamp ($\lambda_{peak} = 365$ nm, Spectronics Corp.) was utilized for the exposure step, during which the polymerizable/cross-linkable materials were photo-chemically transformed. A schematic of the UV-induced polymerization process is presented in the bottom part of Fig. 1. The power intensity of the UV-light incident on the film surface during the photochemical transformation step was measured to be $I = 1.5$ mW/cm², by use of an optical power-meter system (Thorlabs, PM30-120). All of the above device preparation procedures and measurements, except the cleaning of the substrates, were carried out in two interconnected N₂-filled glove boxes ([O₂] < 3 ppm, [H₂O] < 0.5 ppm) with an integrated thermal evaporation unit.

Samples for the Fourier transform infrared (FTIR) interrogations were prepared by mixing the master solutions in a dry mass ratio of poly-PEO:METMA/AMPS:DMPA = 3:1:0.05. This is effectively the same mass ratio as in LEC devices, with the exception that the SY was purposely removed. The carefully stirred blend solution was cast onto silicon substrates, after which the resulting film was dried under vacuum for > 24 h at room temperature. The FTIR spectra were recorded using a Spectrum BX FTIR (PerkinElmer) instrument.

3. Results and Discussion

Fig. 2. A schematic presentation of the UV-activated photochemical stabilization of a doping structure within an LEC device. (a) A pristine ITO/active-material/Al device; (b) the in-situ formation of a light-emitting p-n junction doping structure within the active material following the application of an external voltage; (c) the UV-induced stabilization of the p-n junction structure via activation of the photo-initiator and subsequent cross-linking of the ions and the ion-transport material.

Fig. 2 presents a schematic of the anticipated and desired formation of a photochemically stabilized doping structure within an LEC device. The ITO/active-material/Al sandwich cell was first pre-biased with ITO connected as the positive anode and Al as the negative cathode. During the pre-bias period, ions move to the electrode interfaces to facilitate electronic charge injection and electrochemical doping. At the ITO anode (Al cathode), holes (electrons) are injected onto the conjugated polymer SY. The injected holes (electrons) are subsequently electrostatically compensated by redistribution of AMPS anions (METMA cations) in a process termed p-type (n-type) doping. This doping process transforms the SY into a highly conducting state. With time the doped SY regions grow in size, and after a turn-on time they make contact in the bulk of the active material under the formation of a light-emitting p-n junction, as depicted in Fig. 2(b).

In conventional *dynamic* LECs with mobile ions, the p-n junction doping structure will dissipate when the applied bias is removed, and as a consequence the doping needs to be reformed the next time a voltage bias is applied. This dissipation/reformation process is highly impractical for many applications where a fast turn-on, high rectification of current and light emission, and/or stable response is desired. It is also plausible that the persistent existence of mobile ions within the active material will affect the long-term stability of the device in a negative manner.[44,45] Here, we have attempted to rectify these problems by endowing both the ions and the ion-transport material with cross-linkable units in the form of acrylic double bonds, and – importantly – by including a UV-sensitive photo-initiator into the active material (for chemical structures, see Fig. 1). The idea is to let the ions redistribute and contribute to doping up to the point at which a desired doping structure is attained (e.g., a p-n junction structure as

depicted in Fig. 2b). At this instant, the photo-initiator is activated *via* exposure to UV light, which in turn starts a chain reaction that activates the acrylic carbon double bonds within the ions and the ion-transport material and eventually results in the formation of a cross-linked, and immobile, ionic network. At this point, the device has transformed into a *static* LEC, comprising a p-n junction doping structure stabilized by photochemical means, as depicted in Fig. 2(c).

Fig. 3. FTIR spectra recorded on a {poly-PEO:METMA/AMPS:DMPA} film in the pristine state (bottom black dashed trace ~~marked with solid squares~~) and after UV illumination (top red solid trace ~~marked with open circles~~). The characteristic acrylic carbon double bond vibrations are indicated with blue circles, and the spectrum recorded on the UV-exposed film is shifted upwards for clarity.

In order to evaluate the potential of the above outlined stabilization process, it is educational to perform an FTIR study on a conjugated-polymer-free “active-material” film. If the ionic-network formation process is effective, then the acrylic carbon double bonds within the METMA/AMPS ion pair monomer and the poly-PEO ion-transport material should be transformed into carbon single bonds. This transformation process is conveniently probed with FTIR as the acrylic carbon double bonds -- but not the carbon single bonds -- exhibit characteristic vibrational signatures at 1400 cm^{-1} , 1620 cm^{-1} and 1635 cm^{-1} .^[46]

Fig. 3 presents FTIR spectra of a pristine {poly-PEO:METMA/AMPS:DMPA} film (bottom black dashed trace) and the same film after exposure to UV light for 30 s (top red solid trace). The vibrational fingerprints of the acrylic carbon double bonds are clearly apparent in the pristine film (marked by the blue circles), but completely missing following the UV exposure. This observation strongly indicates that the brief UV light exposure has activated the DMPA photoinitiator, and that the resulting free radicals have started a polymerization/cross-linking process involving a break-up of the acrylic double bonds on the ion-pair monomer and the ion-transport material and a subsequent formation of a desired ionic network.

Fig. 4. (a) The temporal evolution of the current and brightness of an LEC device driven at $V = 10$ V. (b) The initial optoelectronic response for a nominally identical device, with a UV exposure step included between the pre-bias period and the final stabilization period.

Fig. 4 reveals the effects of the UV-exposure step on an actual LEC device by comparison. Fig 4(a) presents the typical optoelectronic response of an ITO/{SY:poly-PEO:METMA/AMPS:DMPA}/Al sandwich cell during the initial operation at $V = 10$ V, but with no UV step included. The increase in current and brightness (up to ~ 3 min) correspond to the formation and development of the p-n junction structure, while the subsequent significant decrease (up to ~ 10 min) can be attributed to various chemical and electrochemical side reactions[45,47] and possibly the installment of steady-state operation[48].

Fig. 4(b) presents the device response of a nominally identical device, but with a 30 s UV illumination period (marked by the hatched area) included at the end of the “pre-bias” period (*i.e.*, the point at which the device performance had reached its peak). During this UV-exposure period, the current is dropping significantly to reach a value of approximately $1/3$ of the maximum value during the pre-bias period. The brightness was not measured during the exposure, but the post-exposure brightness is also significantly lower than the peak value during the pre-bias period. A “stabilization period” of ~ 2 min is observed to follow after the exposure step, during which the current and brightness increase in a relatively concerted manner to reach a steady-state value.

The total measured current in LECs comprises an ionic and an electronic part, where the former dominates during the initial operation[49] and the latter ideally should be solely existent during steady-state operation[48]. The observed drop in current during the UV-exposure step in Fig. 4(b) can be attributed to two different mechanisms: (i) a desired elimination of the ionic conductivity within the active material *via* the ionic-network formation, (ii) a non-desired disturbance of the electronic conductivity (a “side reaction”) within the conjugated polymer. The observation that the brightness has decreased following the exposure implies that the electronic conductivity of the conjugated polymer is partially damaged by the UV-light exposure, as the sole formation of an ionic network should leave the brightness relatively unaffected. The fact that both the current and brightness are observed to increase somewhat during the first ~ 2 min of the stabilization period further suggests that the ionic-network

formation process has not reached completion at the end of the exposure period, and that further cross-linking takes place before a fully stabilized doping profile and steady-state operation can be attained.

Fig. 5. Left graphs: The long-term stability under open-circuit conditions for pre-biased and UV-exposed sandwich-cell devices, with the open-circuit time specified in the inset. Right graphs: The current and brightness rectifying behavior after 24 h of open-circuit storage. The device presented in (a) and (b) was exposed to UV light for 30 s, and the device in (c) and (d) was exposed for 60 s.

Fig. 5 presents results on the long-term stability under idle conditions for pre-biased and UV-exposed ITO/{SY:poly-PEO:METMA/AMPS:DMPA}/Al sandwich cells. Fig. 5(a) shows short-term brightness measurements, following different periods of storage under open-circuit conditions, for a device that had been exposed to UV light for 30 s during the initial operation (see Fig.4b), and Fig. 5(b) shows the current and brightness rectifying behavior of the same device after 24 h of open-circuit storage. Figs. 5(c) and 5(d) present data for an identical device, which had been exposed for twice as long time, *i.e.* 60 s, following the pre-bias operation. Table 1 provides quantitative data for the UV-exposed devices, and also includes results for a control device that had not been exposed to UV-light (see Fig. 4a).

Table 1. LEC performance as a function of UV exposure and storage time.

UV Exposure time (s)	Steady-state brightness at $V = 10$ V (cd/m ²)	Steady-state current efficacy at $V = 10$ V (cd/A)	Retained brightness (in %) following open-circuit storage during:				Current rectification ratio at $V = \pm 15$ V ^b
			2h	6h	12h	24h	
0	87 ^a	1.2 ^a	50	6	0	0	NA
30	100	1.2	97	91	82	60	1000
60	34	0.63	99	97	88	71	1400

^a The device never reached steady-state, and the reported values were measured after 10 min operation.

^b Measured after 24 h of open-circuit storage

It is clear that the UV-light exposure step distinctly improves the stability of the doping within the LEC device. No brightness could be detected from the control device during a short-term measurement following 12 h under open circuit, whereas, *e.g.*, the device that had been UV exposed for 60 s retained 88 % of its initial steady-state brightness following the same storage time. A further demonstration of the existence of a stabilized p-n junction profile within the UV-exposed devices is provided by rectification measurements. When a UV-exposed device was biased with the same polarity as during the pre-bias following 12 h of idleness, it both conducts significant current and emits light; while under reverse bias, the same devices neither conducts nor emits light. This rectifying capacity can be quantified with the current rectification ratio (RR), and the UV-exposed devices exhibit a quite respectable $RR \geq 1000$. The non-exposed device neither rectifies the current nor the light emission. A comparison between the two UV-exposed devices reveals that the shorter exposure time of 30 s results in higher steady-state brightness and larger efficiency, while the longer exposure time of 60 s provides a slight improvement in the long-term stability and in the rectification capacity.

The performance of the photochemically stabilized devices is on par, or better, than previously published chemically stabilized LECs,[39-41] but a comparison with recently published data on conventional dynamic LEC devices based on the same conjugated polymer SY indicates that further improvements are possible.[50-53] In chemically stabilized LECs, with no added initiator compound, the cross-linking of the ions and/or ion-transport material is presumably initiated by the electrons/holes on the conjugated polymer.[39] This has the undesired consequence that the conjugation, *i.e.* the electron transport paths, of the conjugated polymer will be broken at the initiation places. We anticipated that this problem could be resolved by adding a photo-initiator compound to the active material which would function as the initiator site following UV activation. However, the observations that the current and brightness drop markedly following the exposure to UV light and that the steady-state performance is not optimized suggest that further improvements in, *e.g.*, the wavelength and time of UV radiation and the choice and concentration of the photo-initiator compound are necessary, so that direct UV-induced and/or radical-induced damages of the conjugated polymer can be minimized. We hope to be able to come back with the results from such an optimization study in the near future.

We also wish to point out that a photo-activated initiation step brings the possibility of stabilization of an arbitrary doping profile. This is of interest for several reasons. For instance, for many applications it is desirable to attain stabilized doping only at the electrode interfaces and to keep the bulk of the material doping-free. Also, recent data on LEC devices suggest that the fully formed p-n junction structure might not correspond to the best performing device structure.[54, 55] In both cases, it is desirable to stabilize a desired doping structure on-demand, *e.g.*, *via* a short exposure to UV light. The above described challenges and opportunities are planned to be explored in future work.

4. Conclusions

We report a fast and flexible method for the photochemical stabilization of doping structures in conjugated polymer films. By designing an LEC device with an active material comprising a photo-initiator compound, and a cross-linkable ion-pair monomer and ion-transport material, in addition to the conjugated polymer, we show that it is possible to stabilize an electrochemically induced p-n junction doping structure *via* a short-term exposure to UV light. The stabilized junction is manifested in a fast device turn-on and high current and brightness rectification following 1 day of storage under open-circuit conditions. The presented photochemical stabilization method will require further optimization in order to be fully functional, but offers important advantages such as fast execution at room temperature and the opportunity for stabilization of wide range of different doping structures.

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