

Letter

Photo-induced storage and mask-free arbitrary micro-patterning in solution-processable and simple-structured photochromic organic light-emitting diodes



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ABSTRACT

A photochromic diarylethene-based compound BMTA, which undergoes a reversible conversion between ring- open and closed isomers by alternating UV and visible light illumination, has been designed and synthesized. By utilizing a mask-free Digital Micro-mirror Device (DMD) micro-lithography system, arbitrary micro-photopatterning in polymer films doped with BMTA can be easily obtained with UV light writing. This recorded photo information can easily be erased by further visible light irradiation. The reversible and rewritable optical storage is based on photo-switched intermolecular energy transfer between the emissive host and the ring-closed isomer *c*-BMTA. Furthermore, the solution-processable organic light-emitting devices (OLEDs) with the single emitting layer doped with BMTA were fabricated, which exhibit rewritable memory behavior with light control. The luminescence and current density decrease significantly upon UV light irradiation, and recover by further visible light illumination. This is because the hole trapping is much facilitated in closed-ring isomer based devices, due to elevated HOMO level of *c*-BMTA. Without incorporating any cross-linking layer, the maximum luminescence and current density on/off ratios of this solution-processable and simple-structured device are 1.9×10^3 and 1.4×10^2 , respectively. Arbitrary micro-photolithography of OLEDs by DMD system has also been demonstrated, which shows great prospects in large-scale production of high resolution OLED displays.

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1. Introduction

Solution processable organic opto-electronic devices have attracted considerable attention due to their low cost, flexible and large-area production [1,2]. In last decades, remarkable improvements have been obtained in applications of the photochromic materials in opto-electronic devices [3–5], including organic memory diodes [6–8], photochromic light-emitting diodes [9–12], optically switchable multifunctional transistors [13–19], solar cells [20] and electrical circuits [21]. Because these photochromophores undergo a reversible photo-chemical reaction between the open and closed-ring isomers, causing the change of

electrical energy levels. Correspondingly, the absorption and/or photoluminescence spectra, oxidation and reduction potentials, dipole moment, ionization potential (I_p) and carrier mobilities, alter and can reversibly switch [3,22–28]. Among these photochromophores, diarylethenes (DAEs), due to their good thermal stability, fatigue resistance, and easy monitoring by using UV–vis spectroscopy [29–33], are intriguing candidates as the stimuli-responsive materials in solution- and vacuum-processable organic opto-electronic devices [4,6–13,15–17,20,21,28,34].

In recent years, there have been attempts to fabricate memorisable organic light emitting diodes (OLEDs) by utilizing photochromic compounds as an independent light-controlled carrier switch, because the photoirradiation-induced change of I_p affects the current injection into the DAE layer [7,8,10–12]. In the open-ring isomer based device, the barrier for charge injection is large and the current injection is difficult, while in the

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closed-ring isomer based device the photochromic layer exhibits an enhanced I_p , which facilitates the carrier injection from the photochromophore layer to the emitting layer and enables the devices to be lightened. However, to obtain large current on/off ratio in solution-processable OLEDs, an additional cross-linking overlayer onto the PEDOT: PSS layer is generally required [7,11,12], which results in less photo absorption and thus decreased photo-isomerization efficiency. Besides, these cross-linking materials need numerous synthesis and further comprehensive screening efforts in later practical device performances [34]. Moreover, two additional photochromic layer and cross-linking overlayer further complicate the device structure, leading to increased fabrication cost.

In addition, the photo-reactivity of these incorporated photochromophores in OLEDs make the photo-patterning of the light-emitting devices easily available, which can find good signage applications such as warning signs, indicators, decorative light sources, advertising, and logo displays [35–37]. However, this image procedure generally demands irradiation exposure through a specific shadow mask, one-to-one custom-made according to the required graphic pattern, to illuminate the active area. That is, once a new image graphic is acquired, a new shadow mask should be newly customized accordingly. Previously, a technique for arbitrary mask-free micro-patterning in liquid crystal alignment layer and local polarization control for light wavefront have been proposed and implemented, by utilizing a micro-lithography system with a Digital Micro-mirror Device (DMD) [38]. The DMD, utilized as a dynamic virtual “mask”, generates arbitrary patterns by individually tilting angle control of each mirror and thus supplies a low-cost mask-free method for any pattern design.

In this work, without any additional cross-linking layer, solution-processable and simple-structured photochromic OLEDs, with the single emitting layer doped with a DAE-based photochromophore, are easily fabricated and exhibit memory behavior with the maximum on/off ratios of luminescence and current density of 1.9×10^3 and 1.4×10^2 , respectively. Arbitrary mask-free micro-patterning of photochromic OLEDs as well as photochromic polymer emitting films are also demonstrated by utilizing the DMD system, indicating great potentials in low-cost and high-resolution optical memory and OLED displays.

2. Experimental section

o-BMTA has been synthesized according to the previously reported method [39]. UV/Vis absorption and fluorescence spectra are performed on Shimadzu UV-3150 and Shimadzu RF-5301 spectrometer, respectively. Optical irradiation was conducted with a CEL-HXF300 xenon lamp by utilizing color filters (UV: $\lambda < 400$ nm; Vis: $\lambda > 450$ nm). The optimum device configuration in our study is indium–tin oxide (ITO)/poly(3,4-ethylenedioxythiophene)-doped poly(styrene sulfonic acid) (PEDOT:PSS)/(2-Methyl-9,10-bis(naphthalen-2-yl)anthracene (MADN): T4TB: BMTA/TPBi/LiF/Al. A thin hole injection layer of PEDOT: PSS (20 nm) was cast onto transparent conductive ITO at a spin speed of 2000 rpm. After baking at 120 °C for 15 min in an oven, a MADN layer (30 nm) doped with T4TB (1 wt%) and BMTA (1 wt%) was spin-coated on top of the PEDOT:PSS layer from its chloroform solutions. Finally, a 20 nm electron transport layer (TPBi), LiF (0.8 nm) and Al (100 nm) were consecutively deposited by thermal evaporation in vacuum. The luminance–current–voltage characteristics of the devices were recorded using a combination of a source meter (Keithley 2602) and a luminance meter. The EL spectra were determined using a spectrophotometer (Photo Research PR655 SpectraScan). All the devices were characterized without

encapsulation, and all the measurements were carried out in ambient atmosphere. The emission area of the devices is $4 \times 3 = 12 \text{ mm}^2$.

3. Results and discussion

The photochromic compound BMTA, phenylbenzothiazole of diarylethene imide derivative, has been synthesized by condensation of 4,4-(cyclopentene-1,2-yl)-bis(5-methyl-thiophene-2-formaldehyde) and 3-(benzo[d]thiazol-2-yl)aniline (see Scheme S1 and the synthetic details in the Supporting Information). BMTA undergoes a reversible photochromic reaction between the ring-open and closed isomers (see the chemical reaction in Fig. 1a), either in solution or film, with a reversible change of their absorption spectra profiles (see Fig. 1a and Fig. S1). By irradiation with UV light ($\lambda_{\text{irr}} = 200\text{--}400$ nm, $I_0 = 2.49 \text{ mw/cm}^2$), the open-ring isomer (*o*-BMTA) converts into the closed-ring isomer (*c*-BMTA), with the color changing from transparent into blue, accompanied by a newly emerging broad absorption band around 580–590 nm. Upon further visible light illumination ($\lambda_{\text{irr}} > 450$ nm, $I_0 = 2.49 \text{ mw/cm}^2$), *c*-BMTA returns to *o*-BMTA, with the gradual decrease of the 580–590 nm absorption band. Meanwhile, the solution becomes transparent again.

Based on this, photo switchable hybrid T4TB/BMTA (wt%: wt% = 10:1) films can be obtained (Fig. 1b). Upon UV light irradiation, *o*-BMTA converts into *c*-BMTA, and the fluorescence of the hybrid film (peaked around 550 nm) is efficiently quenched, with the fluorescence on/off ratio of 8. Subsequently, by visible light illumination, the emission intensity recovers (Fig. S2). This results from the photo-switched intermolecular energy transfer. The yellow emitting material T4TB obtained in our previous work [40] was chosen as the emissive energy donor, due to good spectral overlap between its emission (peaked around 540 nm) and the newly appeared absorption of *c*-BMTA (peaked around 580–590 nm) (see Fig. 1a), which enables efficient energy transfer (only occurring between the T4TB donor and the acceptor of the closed-ring isomer *c*-BMTA) and thus reversible tuning of emission intensity by alternating UV and visible light illumination.

Thus, by using DMD system, mask-free arbitrary high-contrast micro-images and erasable/rewritable optical information storage have been realized in PVK/T4TB (20 wt%)/*o*-BMTA (4 wt%)/B(C₆F₅)₃ (46 wt%) hybrid polymer film (see Fig. 2). Here, PVK is applied as the polymer host to improve the solution processability and B(C₆F₅)₃ is utilized to cause change of the emission profile with a newly emerging blue fine-structured emission (see Fig. S3a) possibly due to an intermolecular reaction [41] to enhance the imaging contrast (see Fig. S3b). In the DMD computer exposure system [42], the 365 nm UV light can irradiate only through the white regions within the input black-and-white pictures, leading to an emission turn-off in the corresponding area in the film which is caused by the UV-induced energy transfer as discussed above. By utilizing various input electronic pictures in this DMD system, arbitrary complex micro-images can be easily obtained without any shadow mask (Fig. 2). As seen from these images, the resolution can reach 2–3 μm . It is noteworthy that these recorded images can easily be erased upon subsequent visible light irradiation ($\lambda_{\text{irr}} > 450$ nm), and a new micro-patterning can be written by UV light irradiation (not shown). The writing and erasing processes are reversible and repeatable, indicating that these hybrid films are good candidates for application as rewritable high-density optical information storage.

Enlightened by these results, we further expected that the microlithography can also be applied in data storage of photochromic OLEDs. The solution-processable devices were fabricated with configuration of ITO/PEDOT: PSS/MADN: T4TB (1 wt%): *o*-BMTA (1 wt%)/TPBi/LiF/Al (Fig. 3a). Here, MADN doped

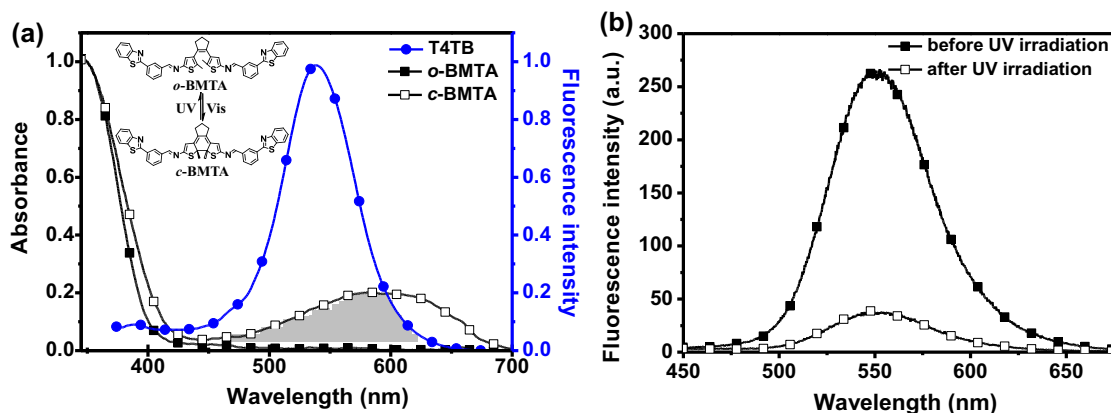


Fig. 1. (a) Normalized absorption of *o*-BMTA and *c*-BMTA, and PL spectrum of T4TB in films; (b) fluorescence spectra of T4TB/BMTA (wt%: wt% = 10:1) hybrid film before and after UV light irradiation (365 nm, $I_0 = 5.86 \text{ mW/cm}^2$, 2 min).

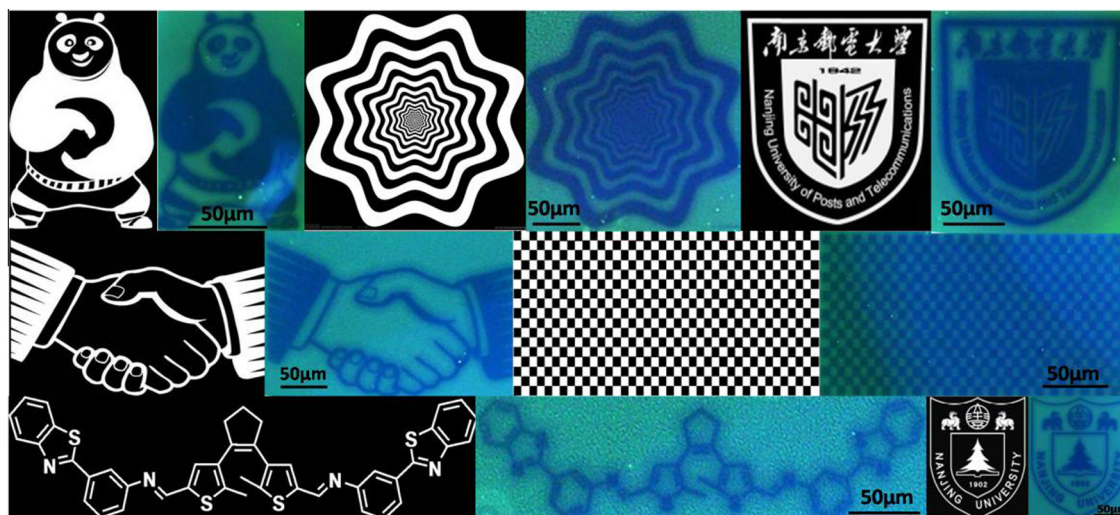


Fig. 2. Fluorescence images (right) on PVK/T4TB/*o*-BMTA/B(C₆F₅)₃ hybrid films by UV light irradiation with utilization of the corresponding black-and-white images (left) under mask-free DMD systems.

by T4TB and *o*-BMTA serves as the single emitting layer, and PEDOT: PSS and TPBi as the hole- and electro-transporting layer, respectively. Whatever images are imputed, the corresponding micro-patterning and optical information storage of the OLED can be easily realized by the mask-free DMD micro-photolithography system. Fig. 3b shows three examples of image processed programmable OLEDs. The areas of these OLEDs are as small as $4 \text{ mm} \times 3 \text{ mm} = 12 \text{ mm}^2$.

After irradiation by UV light, the OLED current density is much lowered (Fig. 3c) from 0.46 to 0.003 mA/cm^2 (measured at 6.2 V). The maximum current on/off ratio is measured as 1.4×10^2 , much higher than that in reported solution-processable OLEDs (either as a distinctive layer or doped in the emitting layer) without cross-linking layer (see the Supporting Information in Ref [11]) and comparable to that of vacuum-evaporated OLED [10]. In the open-ring isomer based device, the turn-on voltage (the voltage required to operate the device at a luminance intensity of 1 cd/m^2) is 6.2 V , and the luminescence increases with the operating voltage. Upon irradiation by UV light, the turn-on voltage increases to 9.7 V , and the device suddenly darkens with the luminance decreasing from 4398 to 45 cd/m^2 (measured at 11.8 V , see Fig. 3d). The maximum luminescence on/off ratio is 1.9×10^3 . Such a high-contrast switch in the device allows for a good strategy for non-destructive readout of the light-written information. Notably, this written information can further be gradually erased upon

subsequent visible light irradiation (see Fig. S4), indicating great potentials in rewritable multi-level memories.

The above described phenomenon is in contrary to Zhang and Meerholz's reported photochromic OLEDs [4–5,7–9], in which the emission boosting is observed after UV light illumination. This difference is caused by their different device structures (ITO/CuPc/DTE/Alq₃/Mg:Al, and ITO/PEDOT:PSS/cross-linkable XT PD/DTE/polyspirofluorene/Ba/Al, respectively), where the photochromophores are utilized as the distinctive light-controlled carrier switching layer. The UV-induced I_p narrowing of the closed-ring photochromophore results in a smaller barrier for charge injection, hence improved device performance.

In our work, the DAE-based photochromophore was doped into the emitting layer in the PLEDs. Herein, we offer a UV-light-driven holes trapping mechanism [43,44] to explain the memory behavior. The energy-levels of the applied materials, in the emissive, hole-transporting and electron-transporting layer, respectively, are depicted in Fig. 4. The whole device exhibits a dominant hole-transporting characteristic. After UV illumination, the HOMO level is enhanced by 1.2 eV from $E_{\text{HOMO}(O)} = -5.8 \text{ eV}$ for *o*-BMTA to $E_{\text{HOMO}(C)} = -4.6 \text{ eV}$ for *c*-BMTA (derived from the Cyclic voltammetry in Fig. S5). Therefore, in the closed-ring isomer based device, the injected holes carriers in MADN and T4TB ($E_{\text{HOMO}} = -5.5 \text{ eV}$) can easily be trapped by HOMO of the *c*-BMTA molecules, leading to the decrease of the luminance and current density. However, in

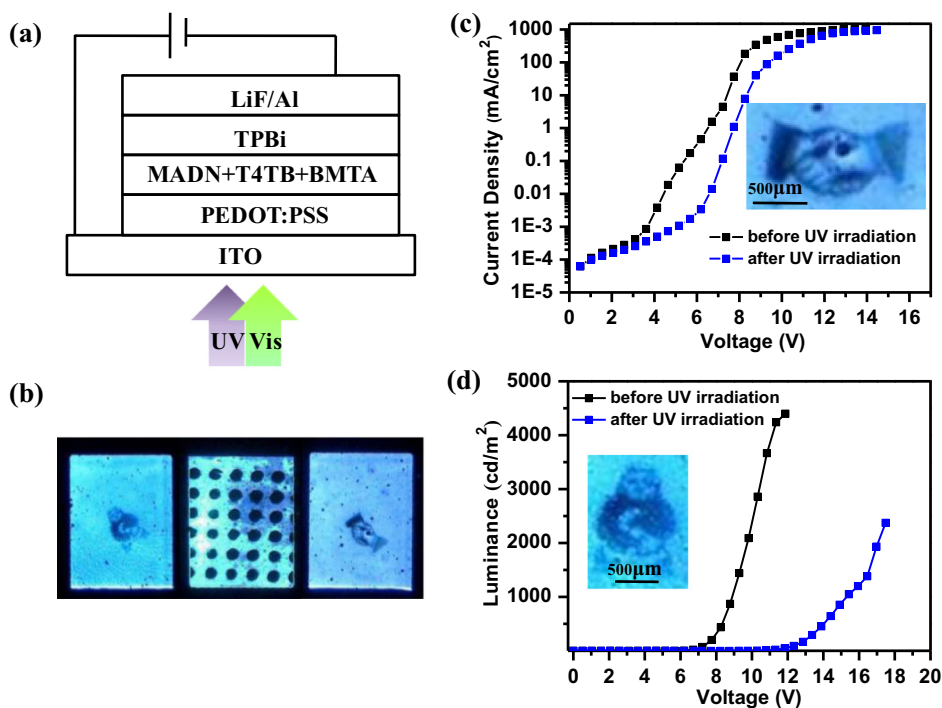


Fig. 3. The programmable OLED structures (a), photo-lithographed images of the emissive OLED (the area is $4 \times 3 = 12 \text{ mm}^2$) at 8.2 V with the DMD system (b); current density–voltage (c) and luminance–voltage (d) characteristics of the devices before and after UV irradiation ($I_0 = 5.86 \text{ mW/cm}^2$, 1 min).

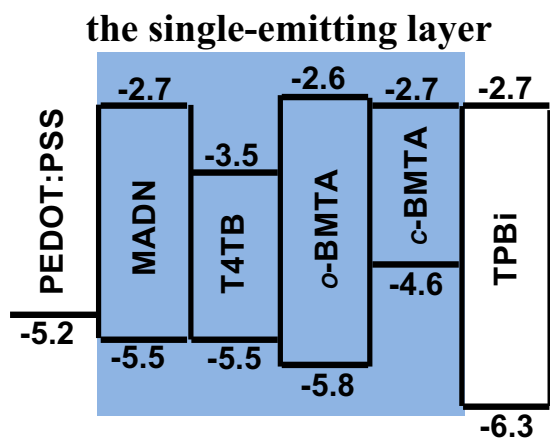


Fig. 4. The energy-levels of each material in the emissive, hole-transporting and electron-transporting layers (the energy levels of MADN, T4TB and *o/c*-BMTA in the single-emitting layer are depicted distinctively for clarification).

the open-ring isomer based device, the hole trapping is not as easy as that in the closed-ring isomer based device, due to lowered HOMO in *o*-BMTA ($E_{\text{HOMO}(\text{O})} = -5.8 \text{ eV}$) than *c*-BMTA ($E_{\text{HOMO}(\text{C})} = -4.6 \text{ eV}$). As a control experiment, we also fabricated the photochromic PLEDs without doping T4TB, with the configuration of ITO/PEDOT:PSS/MADN:BMTA/TPBi/LiF/Al. The maximum current and luminescence on/off ratios are only 14 and 6, respectively (Fig. S6); both are much smaller than those of the T4TB-doped devices. The reason is not clear, and we suppose that this may be related to competitive hole trapping by T4TB and large spectral overlap between T4TB emission and *c*-BMTA absorption.

4. Conclusions

In summary, without utilizing additional photo cross-linking layer, simple-structured solution-processable memorisable OLEDs,

with the single-emitting layer doped by photochromic BMTA, have been demonstrated. This is caused by easier hole trapping due to the elevated HOMO level in the closed-ring photochromic isomer. The maximum on/off ratios of luminescence and current density are 1.9×10^3 and 1.4×10^2 , respectively. Moreover, by utilizing the DMD micro-lithography system, mask-free arbitrary optical polymer emitting films and information storage of both photochromic polymer emitting films and solution-processable photochromic OLEDs have been demonstrated, based on the photo-switchable intermolecular energy transfer and hole-trapping mechanism, respectively. Easy mask-free arbitrary micro-lithography of these solution-processable OLEDs indicates great prospects in large-scale fabrication of high-resolution OLED displays and high-density photo-assisted electrical memories.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.orgel.2015.07.059>.

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