Optical and electrical modulation in ultraviolet photodetectors based on organic one-dimensional photochromic arrays

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Abstract
Organic photochromic materials have drawn considerable attention for their potential applications in large-scale and low-cost optoelectronics owing to unique tunable physicochemical properties. For organic photodetectors, photochromic materials have realized optical and electrical engineering of semiconductor layers, which incorporate not only tunable performance, but also functionalities to optoelectronic devices. However, the essential challenge is to assemble large-area photochromic micro- and nanostructure arrays with controllable geometry and precise alignment, which restricts the integration of multifunctional optoelectronic devices. Herein, we fabricate organic photochromic one-dimensional (1D) arrays via a feasible solution process through the confined crystallization of organic molecules. By modulating and controlling the photoisomerization behaviors, these 1D photochromic arrays possess broad spectral tunability, which ensure tunable photoresponse. Furthermore, we investigate the crystallographic transition and electronic performance variation of these 1D photochromic arrays. By adjusting the dwell time of ultraviolet (UV) irradiation, the UV photochromic photodetectors realize tunable and repeatable responsivity from 85.6 to 0.709 mA/W. Our work...
1 | INTRODUCTION

Stimuli-responsive materials are of particular interest due to their tunable and reversible physicochemical characteristics under external stimuli, such as pH, temperature, electricity, or light. Among them, light is an attractive stimulus for constructing functional devices because of its spatiotemporal controllability. Thus far, extensive efforts have been directed on the integration of photochromic materials into multiple functional devices such as light-emitting diodes, field-effect transistors, and photodetectors. Under light illumination, photochromic materials undergo a distinct conformational transition between two isomers, leading to striking structural variations with different physicochemical properties. Introducing photochromic materials into semiconducting layer to tailor optical and electrical properties has attracted broad interests, due to the potential in responsive optoelectronic devices. The essential challenge for the integration of organic photochromic materials is focused on controlling nucleation and growth to pattern programmable micro/nanostructures with strict position and long-range order owing to the poorly controlled capillary flow in the solution processes. To accommodate this challenge, the surface shearing or templates with asymmetric wettability have been demonstrated to steer the pinning and receding of three-phase contact line of microdroplets for the controllable crystallization of organic molecules.

Ultraviolet (UV) photodetectors based on various organic semiconductors have been achieved for broad applications toward environmental monitoring, biological analysis, image sensing, and optical communication. In particular, UV photodetectors based on photoresponsive materials are highly attractive owing to their direct optical and electrical engineering of semiconductor layers, which have been applied for the multifunctional optoelectronic devices. For optical operation of UV photodetectors, organic photochromic materials exhibit high optical response and tunable photosensitivity, which can be easily harnessed to modulate physicochemical properties by photovoltaic and photoisomerization effects within an individual system. In terms of electrical modulation, remote and accurate photoinduced manipulation is of great importance in effectively operating photodetector properties, such as charge transport, interface trap density and photoresponsivity. Recently, studies mainly focus on the effective optical and electrical modulation within hybrid photochromic units. However, this strategy suffers from the risk of phase separation, inevitable grain boundary and large defect density, which could be addressed by single-crystalline system. Therefore, a versatile platform to obtain high-quality photochromic arrays exhibits strong potential for the integration of UV photodetectors and the development of optical memory devices.

In this study, we fabricated organic one-dimensional (1D) photochromic arrays to modulate UV photodetectors performance with high tunability and controllability. A stilbene oligomer material (ethene-2,1-diyl)-dibenzonitrile (o-BCB) is utilized as the photoresponsive layer, which can undergo photoisomerization between trans and cis isomers that possess tunable optical and electrical structures. By using the asymmetric-wettability assembly method, we controlled the nucleation site and the growth direction of organic 1D o-BCB photochromic arrays with high-quality and strict alignment. We further investigated and modulated gradual conformational transition behaviors within 1D o-BCB photochromic arrays. Moreover, we combined the crystallographic structure of these photochromic microwires with local electrical properties to reveal photoisomerization transition in these 1D photochromic arrays. Based on 1D photochromic arrays, we fabricated UV photodetectors and further explored their photochromic behaviors in devices. Through adjusting the UV irradiation time, the optoelectronic properties of these 1D photochromic arrays can be tuned with tunable photoresponsivity varied from 85.6 to 0.709 mA/W.

2 | EXPERIMENTAL

2.1 | Fabrication of 1D photochromic organic arrays

First, to fabricate 1D trans-o-BCB single-crystalline arrays, o-BCB powders (purchased from Sigma-Aldrich) were dissolved in N,N-dimethylformamide (Sigma-Aldrich; super dry reagent) with the concentration of 5 mg/ml. Then 10 μl organic solution was carefully dropped onto the topographical template with asymmetric wettability and covered by a
flat substrate. The assembly system was placed in a vacuum oven at 80°C for 6 h to totally evaporate the solvent. After total evaporation of solvents and removal of template, the aligned 1D arrays were fabricated on the substrate. To fabricate of 1D photochromic arrays, a UV LED light (M365LP1; Thorlabs) was used as photoradiation source (365 nm, 76.5 mW/cm²). 1D trans-o-BCB single-crystalline arrays were directly exposed to the UV radiation, then 1D photochromic crystal arrays at different time were obtained.

### 2.2 | Characterizations

The fluorescence microscope images were captured on an optical microscope (Vision Engineering Co.) coupled with a CCD camera (DP80; Olympus). The morphological characterization of 1D arrays was achieved by scanning electron microscopy (SEM; S-4800; Hitachi) at an accelerating voltage of 5.0 kV. The transmission electron microscopy (TEM) characterization was carried out on JEM-2100 operating at an accelerating voltage of 100.0 kV. The energy levels of different photochromic arrays were collected by ultraviolet photoelectron spectroscopy (UPS; ESCALAB 250Xi; Thermo Fisher Scientific) with a 21.22 eV light source. The UV-vis absorption spectra were conducted under a UV‐Visible/NIR Spectrophotometer (Varian Cary 5000). The photoluminescence (PL) emission and Raman spectra of photochromic arrays were recorded on microscopic confocal laser Raman spectrometer (Renishaw inVia‐Reflex). The temperature-dependent PL mappings were collected on Horiba FluoroMax-4-NIR spectrophotometer. The atomic force microscopy (AFM) and conducting AFM (C-AFM) measurements were taken on an atomic force microscope (Dimension Icon; Bruker) equipped with TUNA module using contact mode. Noteworthy, the C-AFM measurements were carried out at a bias of −5 V. The X ray diffraction (XRD) diagrams were collected on a powder X ray diffractometer (D8 focus; Bruker) equipped with Cu Kα radiation (λ = 1.5406 Å). To quantify the crystallographic transformation of photochromic microwires, grazing incidence wide-angle X ray scattering (GIWAXS) was employed on Beijing Synchrotron Radiation Facility with an incidence angle of 0.2°.

### 2.3 | Photochromism-based photodetector fabrication and electronic measurement

The 300 nm Si/SiO₂ substrates were treated with ethanol, acetone and isopropanol, then dried under a stream of nitrogen. The 1D photochromic array photodetectors were fabricated on Si/SiO₂ substrates by thermally evaporating Cr/Au (10 nm/100 nm) as electrodes. The effective area of devices is around 20 μm² for 4–5 organic microwires. The channel width and length were 40 and 10 μm, respectively. The electrical properties of photodetectors were analyzed by utilizing a Keithley 4200 semiconductor characterization system with a manual probe station (Lake Shore) at the room temperature in a vacuum of 9.5 × 10⁻⁵ mbar. The light irradiance for the photodetection measurement was a LED light (λ = 365 nm). The light illumination intensity was tuned by a LED controller (DC2200; Thorlabs) and calibrated by a silicon photodiode (S130C; Thorlabs). The temporal response performance was measured by a mechanical chopper at the illumination power of 22.3 mW/cm² with 30 V bias.

### 3 | RESULTS AND DISCUSSIONS

To obtain o-BCB single-crystalline arrays, we utilized a solution process by manipulating capillary bridges within an asymmetric-wettability assembly system, including a flat substrate, a droplet of organic solution and an asymmetric-wettability topographical template (see details in the Experimental Section). The asymmetric-wettability micropillar-structured templates were acquired by selective modification with heptadecafluorodecyl-trimethoxysilane (FAS) molecules (see details in Supporting Information Note S1 and S2). After modification, the water contact angles (CA) in different regions were measured as 105.3 ± 2.7° at sidewall and nearly 0° at tops (Figure S1). As the schematic illustrations shown in Figure S2, the asymmetric-wettability assembly system evolved from a confined liquid film via a series of discrete capillary bridges to aligned organic arrays. We also captured in situ fluorescence micrographs to further study this dewetting process. First, the whole assembly system originated from a continuous film confined between the superlyophilic micropillars' tops and a flat substrate. Owing to the wettability diversity between the micropillars' sidewalls and tops, the organic liquid was confined in the spatial gap between the micropillars. With the evaporation of solution, liquid film then divided into various discrete capillary bridges, providing the confined space for the nucleation and growth of organic crystals. Ultimately, precisely positioned and highly aligned 1D arrays were fabricated on the substrate after the total evaporation of organic solution.

To identify the morphology and crystal structure of organic 1D o-BCB arrays, we presented the SEM and TEM. As shown in Figure 1A, SEM image exhibits high alignment, precise position and uniform morphology of these organic crystals. The high quality of as-prepared organic arrays with limited grain boundary can be further demonstrated by the zoom-in SEM image and...
TEM image (Figures S3 and 1B). Selected area electron diffraction (SAED) pattern with sharp diffraction spots corresponding to the crystal faces of (100) and (020) indicates that 1D o-BCB single crystals grow along with (100) crystallographic orientation. Figure S4 presents the SAED patterns of different positions in 1D structure, confirming the single crystal state of 1D o-BCB arrays. Furthermore, we carried out parallel- and cross-polarized reflection fluorescence images of 1D arrays, demonstrating the pure orientation of 1D o-BCB arrays (Figure 1C). The angle-dependent fluorescence intensity also depicts the highly crystallographic alignment of these 1D o-BCB single-crystal arrays (Figure 1D).

As observed in pioneer investigations, the trans-configuration of o-BCB can convert to the cis-configuration under UV irradiation, owing to the torsion of o-BCB molecular configuration (Figure S5). As shown in Figure 2A,B, after directly exposure under a UV light-emitting diode (LED) light (365 nm, 76.5 mW/cm²) for 40 min, the fluorescent color of photochromic microwires changed due to the trans/cis transition of o-BCB molecules. To tune optical photochromic behaviors of these 1D photochromic arrays, both qualitative and quantitative analyses were introduced. We first carried out the UV-visible absorption and PL spectra (Figure S6) on quartz substrates during the UV illumination with the time interval of 10 min. The intensity of the absorption peak at λ = 280 nm increased and at λ = 360 nm decreased under continuous UV illumination, indicating the trans-to-cis conformational change. Fluorescence emission spectra of photochromic microwires also illustrated this gradual conformational transition process. The PL emission peak varied from 500 to 420 nm with the elongation of UV illumination time. Noteworthy, optical spectra measured before and after 40 min irradiation, indicating that the maximum transition of these photochromic microwires were obtained. As provided in Figure S7, we also employed the solution emission spectra and found the reduction of the integrated PL intensity under continuous UV illumination.

Theoretical studies of trans and cis o-BCB molecular configurations energy levels were employed through a density functional theory method (Supporting Information Note S3). Figure 2C exhibits the energy levels of the ground states (S₀) and excited states (S₁) of the trans and cis molecular configurations. The trans-o-BCB is excited from the ground state to the excited state. The geometry then changes to the conical intersection (CI) region via
vibrational relaxation, and finally goes back to $S_0$ state to the cis configuration. It is also shown that the $S_0$ energies of the two configurations are very close, while the $S_1$ states separate much more. Moreover, the cis-o-BCB configuration loses its planarity with an angle between two benzene rings being ca. 51°, which can be explained with the torsion of molecular geometry. A detailed experimental study of the energy level structure was obtained by UPS in Figure S8. The highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) of 1D trans-o-BCB arrays are $-6.14$ and $-2.70$ eV, respectively. Moreover, the mixed trans/cis 1D arrays upon 40 min of UV illumination present the HOMO of $-6.14$ eV and LUMO of $-1.71$ eV, respectively, which is in consistency with our theoretical calculations.

As the PL decay shown in Figure S9, initial organic microwires exhibit PL lifetime of 9.6 ns, which is much higher than these microwires (1.4 ns) after being exposed to UV light for 40 min. Figure S10 shows the raman scattering spectra of photochromic microwires with the dwell time of UV illumination increasing. The weakened Raman peak at 1180, 1210, and 1590 cm$^{-1}$ unambiguously demonstrates the structural transition. Furthermore, the drop of integrated Raman intensity resulting from the low crystallinity can be elucidated via the crystallographic transition properties. To further explore the mechanism of the photoisomerization of trans/cis o-BCB isomers, the temperature-dependent PL mappings were recorded tuning temperature from 5 to 295 K (Figure 2D–F). At the initial state, the main PL emission peak lies at 520 nm. After UV irradiation for 20 min, a peak at 445 nm appears, suggesting the presence of cis-o-BCB isomers. Furthermore, the progressive increasing around 445 nm indicates that the trans-to-cis conformational transformation proceeds and the cis-o-BCB isomers gradually dominate within these photochromic microwires after 40 min irradiation.

Further investigation of the crystalline transition structure of photochromic microwires was employed by XRD and GIWAXS. Crystallography structures of 1D trans-o-BCB arrays were collected by XRD with a series of diffraction peaks at the $2\theta$ of 6.04°, 11.52°, 17.22°, 23.08°, 28.92°, and 34.81°, which could be ascribed to the (001), (002), (003), (004), (005), and (006) planes, respectively, indicating that the fabricated 1D arrays sustain high crystallinity and pure crystallographic orientation. With the increasing time of UV illumination, we observed the reduction of the integrated XRD diffraction peaks, suggesting the trans-to-cis transition of the o-BCB molecules. After 40 min of UV illumination, 1D photochromic arrays showed almost no signal in diffraction peaks (Figure 3A). GIWAXS was harnessed to further analyze the crystallographic information of 1D arrays, which enabled us to better identify the crystalline transition structure (Figure 3B–D). At the original state, strong out-of-plane (001) diffractions were observed, confirming the pure (100)-orientated trans-o-BCB single-
crystalline microwires. After 20 min of UV illumination, less signal could be measured in the diffraction of (001) compared with the initial state. Moreover, (011) and (101) diffractions still remained, which indicated the maintaining of (100) crystal orientation. Diffraction patterns of 1D photochromic arrays were gradually attenuated with the dwell time of the UV irradiation increasing, illustrating the destruction within the crystalline structures in accordance with the weaken of the integrated diffraction peaks measured in XRD diagrams. These results illustrate that the crystalline transition induced by trans-to-cis isomerization is a stepwise process. Based on these evidence, the crystalline transition structure diagram is illustrated in Figure 3E.

Meanwhile, we monitored morphological changes along with crystalline transition of these 1D photochromic arrays. Optical (Figure S11) and fluorescence (Figure S12) images were captured after UV illumination for 0, 10, 15, 20, 30, and 40 min, respectively. Different colors in optical micrographs varied from red to blue, which indicated the decreased height of the photochromic microwires. Furthermore, as seen in Figure S13, organic microwires have
the homogeneous width of ca. 1.8 μm during the UV illumination. AFM images of individual photochromic microwires (Figure S14) with heights of 344.7, 325.8, 317.3, 315.2, 303.3, and 242.7 nm correspond to the different UV illumination durations of 0, 10, 15, 20, 30, and 40 min, respectively, which is in consistency with spectrographic gradient from red to blue observed in bright-field micrographs as mentioned above.

To further discuss the photoisomerization of 1D arrays, C-AFM was carried out to investigate the surface currents of photochromic microwires at different UV irradiation durations. AFM topographical images at 0, 20, and 40 min UV illumination with the heights of 346.1, 320.6, and 243.8 nm corresponded to the C-AFM surface currents of 30.6, 14.7, and 2.70 pA, respectively (Figure 3F–H). The surface roughnesses of these photochromic microwires determined by root mean square on region of 1 × 1 µm² upon continuous UV illumination for 0, 20, and 40 min corresponded to ca. 2.8, 3.5, and 5.6 nm, respectively. The increase of roughness attributes to the increased transition ratio of cis-o-BCB configuration, which could be induced by repulsion of the two phenyl planes. Moreover, these molecular-configuration transitions can not only cause a progressive decrease in electrical conductivity but also lead to a large effect in modulating carrier transport, which will influence the device performance. In conclusion, the o-BCB molecular conformation transition occurs layer by layer on the surface of microstructures, which can be due to the following reasons: (i) our highly-assembled confinement architectures of 1D crystals limit molecular conformation transition inside crystals; (ii) the trans-to-cis isomerization is restrained on the surface layer owing to the 1D single-crystalline arrays providing less interspace for spatial transition changes. Here, our experiments also demonstrate that trans-to-cis isomerization of as-fabricated photochromic microwires could be an irreversible transition process through keeping the sample in the dark condition for two weeks and subsequently heating at 100°C for 1 h (Figure S15).

Based on the properties of o-BCB photoisomerization, we fabricated 1D photochromic array photodetectors to elucidate and modulate the optoelectronic behaviors. As presented in the schematic illustration of a typical structure of 1D array photodetectors (Figure 4A), we applied a bias voltage on a pair of pre-sputter Cr/Au (10 nm/100 nm) electrodes (see details in Section 2). Typical fluorescence microscope and SEM images of the photochromism-based devices show microwires structure between two electrodes with a channel length of 10 μm (Figure S16). All devices on the Si/SiO₂ (300 nm) substrate were measured at dark and 365-nm light illumination by scanning bias voltage from −30 to 30 V. It is worth pointing out that the measured condition of

![Figure 4](image-url)
365-nm LED light with a maximum intensity of 22.3 mW/cm² is too weak to induce significant configuration transition (Figure S17).

To discuss the photoisomerization in optoelectronic properties of these photochromic microwires, we quantify the transition ratio of cis-o-BCB isomers at different durations of UV irradiation based on the analysis of the absorption spectra (Figure 4B). As shown in Figure 4C, photochromism-based devices exhibit dark currents from $1.10 \times 10^{-12}$ to $3.03 \times 10^{-14}$ A and photocurrents from $2.47 \times 10^{-11}$ to $8.83 \times 10^{-14}$ A at the UV illumination durations range from 0 to 40 min. Average dark and photo currents of photochromic microwires were determined by three sets of electrical measurements presented in Figure S18. After 40 min of UV illumination, both the photo and dark currents degrade by two orders of magnitude, corresponding to the maximum transition of trans to cis configuration. The reduction of dark and photo currents values demonstrates the inefficiency of photogenerated carriers and charge flowing after the photoisomerization, which coincides with the C-AFM studies mentioned above.

Figures 4D and S19 provide the detailed plots of the device performance of 1D photochromic arrays under UV illumination with different intensities. First, the photoresponsivities of the initial 1D trans-o-BCB arrays were assessed by the I-V measurements under dark and different light illumination with the power intensity ranging from 0.0763 to 22.3 mW/cm². With the increasing of light input intensity, we observed excellent linear response behaviors resulting from the generation of photocarriers, demonstrating the filling of traps and enhancement of carrier recombination under high irradiance. With the dwell time of 20 min under UV illumination, we measured the photocurrents of the devices as $5.00 \times 10^{-13}$ A under lower incident intensity of 1.28 mW/cm². When the photoisomerization in 1D arrays was nearly saturated after 30 or 40 min of UV irradiation, the linear response behaviors were lost. Figure 4E plots the illumination power dependent responsivities upon different photoisomerization dwell time. Responsivity, R, can be calculated by the following equation: $R = (I_{\text{illuminated}} - I_{\text{dark}})/P$, where $I_{\text{dark}}$ and $I_{\text{illuminated}}$ are currents under dark and light illumination, respectively, and P is the illumination power. We can obtain the modulated photochromism-based photodetectors with responsivities of 85.6, 14.3, and 0.709 mA/W, for isomerization time of 0, 10, and 20 min, respectively.

Another crucial parameter for determining the sensitivities of UV photodetectors is their temporal response. The I-t curves of our 1D array photodetectors in Figure 4F were also characterised at a bias of 30 V with the 365-nm LED intensity of 22.3 mW/cm². As the 365-nm LED light is turned on and off, the photocurrents of 1D photochromic arrays increase to 19.0, 8.58, and 2.78 pA and decrease to 1.69, 0.875, and 0.401 pA at the photoisomerization time of 0, 10, and 20 min, respectively. The controlled on/off response behaviors claim the charge transport tunable properties based on photochromic array photodetectors. After placing in the dark condition and thermal heating (Figure S20), the mixed trans/cis organic array photodetectors modulated by the UV irradiation were maintained, indicating the stability of our fabricated devices.

4 CONCLUSIONS

In summary, we have demonstrated the controlled optical and electrical modulation in UV 1D photochromic photodetectors. Based on high-quality organic single-crystalline arrays, we further investigate and modulate the optical, crystallographic, and electrical transition properties within these photochromic o-BCB microwires. The photodetectors based on 1D photochromic arrays exhibit modulated performance including high photoresponsivity and stability through adjusting different UV irradiation durations. Our assembly system of organic 1D photochromic arrays holds potential perspectives for integrated applications of nonvolatile memory and multifunctional optoelectronic devices.

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CONFLICT OF INTERESTS

The authors declare no conflict of interest.

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**SUPPORTING INFORMATION**

Additional Supporting Information may be found online in the supporting information tab for this article.

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