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**ELECTRICALLY RECONFIGURABLE LIQUID  
CRYSTALLINE MIRRORS (POSTPRINT)**

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**AFRL/RX**

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Interim Report**

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# Electrically Reconfigurable Liquid Crystalline Mirrors

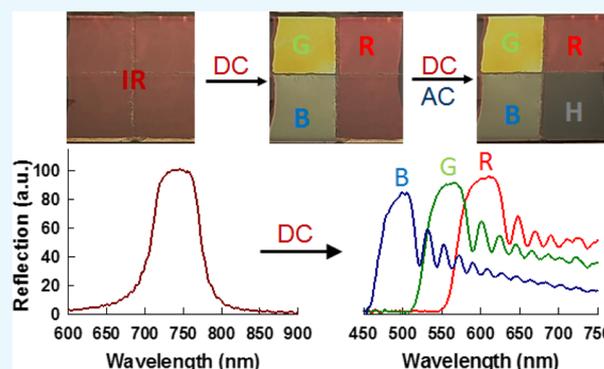
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**S** Supporting Information

**ABSTRACT:** Reconfigurable optical materials are critical to realizing light control in eyewear or architectural windows. Here, we report on the electrical reconfiguration of the selective reflection of cholesteric liquid crystals (LCs). The distinctive responses detailed here are enabled by the preparation of a structurally chiral polymer stabilizing network that enforces anchoring of a low-molar-mass liquid crystalline media with positive dielectric anisotropy. The pitch of the reflective optical elements is directly regulated by a dc field, resulting in red or blue reflection wavelength tuning or broadening. The use of the positive dielectric LC host in concert with optimization of the material preparation conditions allows for reorientation of the LC molecules to achieve an optically clear state (homeotropic orientation) by the application of an ac field. In this way, the selective reflection of the optical elements can be moved, widened, and turned on and off. The electro-optic characteristics of these materials are another step forward to enabling the use of these materials in optics and photonics.



## INTRODUCTION

Reconfiguration of the optical properties of materials enables functional performance benefits in consumer goods, automobiles, and aerospace systems. Dynamic changes to optical material properties including absorption, diffraction, reflection, and scatter have been the subject to intense research. Various stimuli are capable of triggering optical reconfiguration of these properties including heat, light, chemical, and electric field. Chromic devices (photochromic and electrochromic) are increasingly widespread in light control applications as wearables or in windows.

Liquid crystalline materials, pervasive in the display industry, are synonymous with stimuli-induced reconfiguration of optical properties. Of particular interest is the inherent and selective reflection of the cholesteric liquid crystal (LC) phase.<sup>1,2</sup> This phase forms naturally in chiral liquid crystalline molecules but is more commonly formulated via chirality transfer from a dopant to a nematic LC host.<sup>3–6</sup> In this way, the spectral properties of this phase are governed by the composition of the formulation and can be readily adjusted to prepare optical elements across the electromagnetic spectrum (UV to long wavelength infrared, ~300–10 000 nm).

Considerable research has examined thermal,<sup>7–12</sup> photonic,<sup>13–15</sup> and electrically<sup>16–19</sup> induced adjustment to the optical properties of cholesteric LCs. A significant fraction of these prior research efforts has focused on electrically induced switching between a transparent and scattering state, commonly referred to as a polymer-stabilized cholesteric texture.<sup>5,6,20–26</sup> These devices switch from an optically transparent state into a

scattering state by the application of an electric field, which orients the LC into a focal conic texture.

Here, we focus on dynamic reconfiguration of the reflection band gap of polymer-stabilized cholesteric liquid crystals (PSCLCs). Recent research has reported the ability to switch the reflection on or off,<sup>27</sup> tune,<sup>28–31</sup> or broaden<sup>3,19,32,33</sup> the selective reflection of these materials. Of relevance to the work detailed here, we have reported on the ability to electro-mechanically distort a polymer stabilizing network incorporated in the material system that directly affects the pitch of the low-molar-mass cholesteric LC phase.<sup>32–34</sup> Put succinctly, the polymer stabilizing network retains a structural chirality derived from the preparation conditions. To date, these effects have been consistently observable only in compositions prepared with negative dielectric anisotropy ( $\Delta\epsilon < 0$ ) nematic LCs. Accordingly, upon the application of a dc field, the liquid crystalline host does not initially reorient to the field direction enabling an ion-mediated distortion of the structurally chiral polymer stabilizing network to act and enforce pitch changes observable as bandwidth broadening,<sup>3,19,32,33</sup> red-shifting tuning,<sup>28–30</sup> blue-shifting tuning,<sup>31</sup> and scatter.<sup>27</sup>

Here, we report the refinement and optimization of the materials chemistry to realize complete optical reconfiguration of the reflection band gap of polymer-stabilized cholesteric LCs in which the reflection notch can be tuned or broadened as well as

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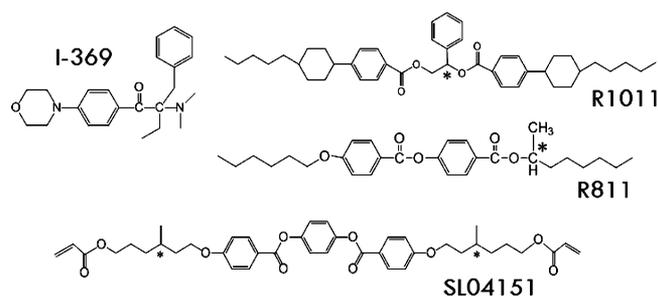
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switched on and off. The switchability of this material system is enabled by the use of conventional, positive dielectric anisotropy nematic LCs ( $\Delta\epsilon > 0$ ). In the applied dc field strengths examined here, the polymer stabilizing network is sufficiently strong to enforce and retain the coupling of the alignment of the low-molar-mass LC to the structural chirality of the polymer stabilizing network. Application of a strong ac field can overcome the favorable interaction of the LC and the polymer network, aligning the LC into the homeotropic orientation. We demonstrate pixelated addressability in which four pixels can be distinctly reconfigured to exhibit an optically clear region adjacent to pixels that exhibit red, green, and blue reflections.

## RESULTS AND DISCUSSION

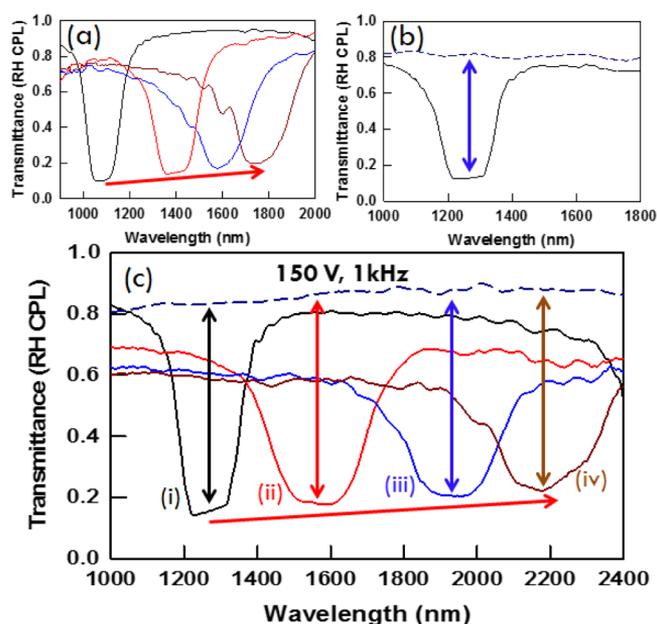
The material systems examined here is formulated with the chiral dopants and chiral LC monomer (SL04151, AlphaMicro, Inc) illustrated in Scheme 1. Samples were prepared over

**Scheme 1. Chemical Structures of the Photoinitiator (Irgacure 369), Chiral Dopants (R1011 and R811), and a Chiral LC Monomer (SL04151) Employed in This Examination**



a range of monomer concentrations based on either a negative dielectric nematic LC host MLC-2079 (Merck) or a positive dielectric nematic LC host E7 (Merck). Polymer stabilization was photoinitiated by Irgacure 369 (BASF). Evident in Figure 1a, in a composition containing 6 wt % SL04151 employing the positive dielectric LC host E7, a large magnitude red shift in the selective reflection of the cholesteric LC is observable upon the application of a dc field. At the appropriate ac field strengths necessary to overpower the alignment of the low-molar-mass LC to the structurally chiral polymer network, the reflection can be removed (Figure 1b). Complete and repeatable switching was observed by the application of a 150 V (1 kHz) ac field with concurrent application of a dc bias, yielding an optical element in which the reflection can be tuned and rapidly switched on and off.

Electro-optic control of the orientation of the LC molecules is the basis of modes widely used to regulate the transmission of light through color filters to yield full color and high-resolution displays. In Figure 2, a material composition is formulated and prepared to exhibit blue-shifting tuning<sup>31</sup> of the reflection. An alignment cell was constructed with four pixels, locally addressable by a voltage source. The selective reflection of the optical element is locally reconfigured from the infrared (IR) (Figure 2a) to exhibit a single red pixel (Figure 2b), red and green pixels (Figure 2c), red, green, and blue pixels (Figure 2d), and an optically clear (homeotropic), red, green, and blue pixels (Figure 2e). As in Figure 1, the local control of the reflection color is governed by the magnitude of the dc field

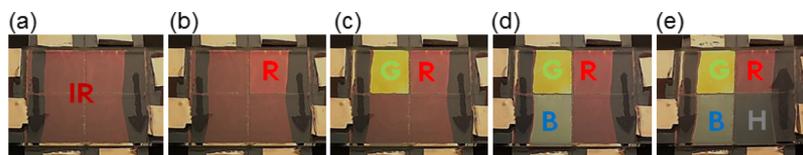


**Figure 1.** (a) Red-shifting tuning of the selective reflection of a PSCLC by the application of dc voltage of 0, 30, 50, and 60 V dc. (b) Reflection switching of the PSCLC band gap is induced by the application of 150 V ac. (c) Reconfiguration of the selective reflection (tuning and switching) is illustrated in sequential transmission spectra (i) 0, (ii) 40, (iii) 60, and (iv) 70 V dc. Reflection switching of (i–iv) was included with 150 V ac (1 kHz). See Movie 1 in the Supporting Information. The sample was formulated by mixing 0.4 wt % I-369, 6 wt % SL04151, 3 wt % R1011, and 90.6 wt % E7 and prepared by exposing to 100 mW/cm<sup>2</sup> 365 nm wavelength UV light for 3 min.

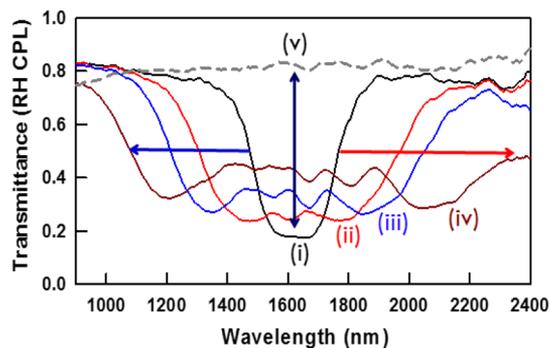
strength and the switchability is enabled by the overlay of a 150 V (1 kHz) ac field to induce a homeotropic orientation. Upon the removal of the field, the local optical properties within a pixel restored to the original selective reflection.

Adjusting the preparation conditions (see Experimental Section) to prepare the PSCLCs examined here and in our prior reports distinguishes the electro-optical output. By reducing the photoinitiator concentration and preparing with a 3 min exposure of 100 mW/cm<sup>2</sup> UV light, the PSCLC optical element now allows for electrical control of the bandwidth (Figure 3i–iv).<sup>3,19,32,33</sup> Here, the application of 20 V dc (1.33 V/ $\mu$ m) causes a sevenfold increase in the reflection bandwidth. Once again, overlaying a 250 V (1 kHz) ac field allows for switchability of the electrically induced broad reflection (Figure 3v). This device is reticent to the use of electrochromic materials in autodimming mirrors but switches faster with greater dexterity in color control.

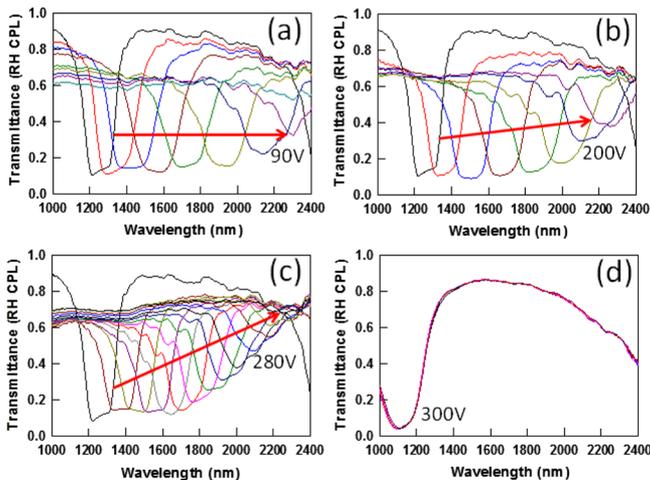
The three examples hereto are based on a polymer stabilizing network composed of 6 wt % monomer concentration, initiated with a 0.4–1.5 wt % photoinitiator, and subjected to various UV exposures (intensity and duration). We conclude this study by illustrating the critical role of monomer concentration in realizing the electro-optic reconfigurability reported here. As evident in Figure 4, increasing the concentration of the chiral liquid crystalline diacrylate monomer SLO4151 (Scheme 1) reduces the magnitude of the response and eventually mutes any sensitivity of the material system to an applied dc or ac field. Evident in the results in Figure 4, the strength of the polymer stabilizing network strongly influences the sensitivity of the optical element to the magnitude of the applied electric field. Also evident in Figure 4d, the formation of robust, highly



**Figure 2.** Control of color in PSCLC optical elements separated into four addressable pixels: (a) 0 V (IR), (b) 3 V dc was applied to the upper right pixel (red), (c) 6 V dc was concurrently applied to the upper left pixel (green), (d) 9 V dc was concurrently applied to the bottom left pixel (blue), and (e) 250 V ac (1 kHz) was applied to the bottom right pixel (homeotropic). The blue-shifting sample was formulated by mixing 1.5 wt % I-369, 6 wt % SL04151, 5 wt % R1011, 5 wt % R811, and 82.5 wt % E7 and prepared by exposing to 250 mW/cm<sup>2</sup> UV light for 30 min.



**Figure 3.** Concurrent switching and broadening of the selective reflection of a PSCLC optical element. (i) 0 V dc, (ii) 5 V dc, (iii) 10 V dc, (iv) 20 V dc, and (v) application of 250 V ac at 1 kHz switches the alignment of the PSCLC to the homeotropic orientation from any of the states (i–iv), removing the selective reflection. This sample was formulated by mixing 1.5 wt % I-369, 6 wt % RM82, 3 wt % R1011, and 89.5 wt % E7 and prepared by exposing to 150 mW/cm<sup>2</sup> UV light for 3 min.



**Figure 4.** Red shift in the selective reflection of the PSCLC optical elements with various polymer concentrations: (a) 6, (b) 8, (c) 10, and (d) 30 wt % over the range of dc voltages up to 300 V.

cross-linked polymer networks at large monomer concentrations prevents an observable optical response.

The electro-optic response of the samples discussed hereto is summarized and contrasted in Figure 5. Upon preparation, the PSCLCs exhibit uniform distribution in the pitch across the sample thickness (previously measured via confocal microscopy<sup>28</sup>) (Figure 5a). From the electro-optic responses evident in Figures 1–4, the deformation of the polymer network to an applied field is strongly dependent on the preparation conditions of the samples, as detailed in a parallel report.<sup>31</sup> It should be noted that in all cases detailed here, the optical

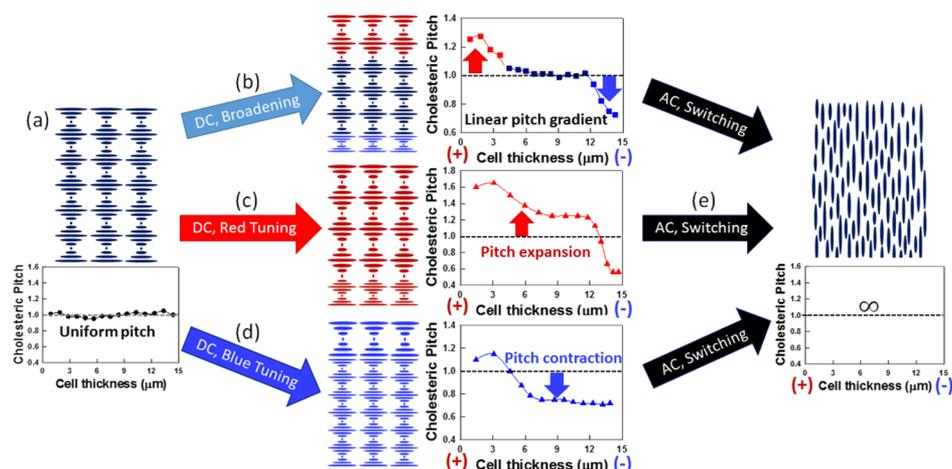
properties of the PSCLC return to the original selective reflection upon removal of the applied electric field.

In the results detailed in Figure 3, bandwidth broadening is observable when the pitch is distorted nonuniformly across the sample thickness. Evident in the illustration and prior characterization via confocal microscopy,<sup>33</sup> the local pitch is expanded near the positive electrode, whereas the pitch near the negative electrode is compressed. The structurally chiral polymer network in the case of the red- and blue-shifting elements is distinguished. In samples that exhibit red shift, as illustrated and confirmed in the confocal examination of pitch,<sup>33</sup> while the pitch is expanded near the positive electrode and contracted near the negative electrode, the pitch in the central 6–12  $\mu\text{m}$  region of the sample is increased uniformly. Conversely, adjusting the preparation conditions such that the samples are prepared with extensive UV irradiation so as to induce blue shifting tuning, the average pitch is reduced by deformation, yielding a blue shift in the selective reflection.<sup>31</sup>

The sensitivity of the electro-optic response to the preparation conditions and photoinitiator concentration have been detailed in a recent report.<sup>31</sup> Summarized here, photopolymerization of the diacrylate monomers reaches full conversion in less than a minute of exposure. However, evident in this report and our prior studies, extended UV irradiation differentiates the electro-optic response when photopolymerization is initiated with aminoketone photoinitiators such as Irgacure 369. We have hypothesized that this associated with the postfunctionalization (reaction) of the residual initiator and the polymer network. The morpholino group of Irgacure 369 is known to be an efficient trap for cations, increasing the sensitivity of the polymer network to ionic interaction. Further, heterogeneous distribution of these fragments across the sample thickness could yield heterogeneous deformation that could explain the nuanced electro-optic outputs we have observed.

## CONCLUSIONS

This report details complete control of the spectral reflection of cholesteric LCs, enabled by the optimization of material concentration and preparation that allows for these devices to be based on positive dielectric anisotropy LCs. Preparing these optical elements on this type of LC allows for them to be reoriented to an applied ac field. Accordingly, the optical elements have distinctive functional performance wherein the presence, position, and breadth of the selective reflection can be reconfigured. The dynamic control of the spectral content of these materials can also be spatially manipulated. The material system reported here extend and open up new application avenues for these materials spanning aerospace, consumer goods, architecture, and sensing.



**Figure 5.** Summary of the results presented here. (a) PSCLC samples are uniform in pitch length across the 15  $\mu\text{m}$  thickness. (b) For samples that exhibit bandwidth broadening, the structural chirality of the polymer stabilizing network is linearly deformed across the sample thickness, yielding a continuous distribution in pitch length. The pitch compresses near the negative electrode and expands near the positive electrode. (c) For red-shifting tuning, the average pitch length in the sample thickness is increased by the application of a dc voltage. (d) When the materials chemistry has been adjusted via long duration UV exposure, the materials exhibit blue-shifting tuning where the average pitch length is shortened. (e) Regardless of the nature of the structural chirality and the response to an applied dc field, overlaying an ac field of sufficient strength can enforce reorientation of the LC molecules into the homeotropic orientation (pitch length goes to infinity) into an optically clear state. Removing the ac field either restores the elements to the (a) 0 V state or to the (b–d) specific optical output caused by the underlying dc field.

## EXPERIMENTAL SECTION

**Preparation of PSCLCs.** Alignment cells were prepared from indium tin oxide-coated glass slides (Colorado Concepts). The substrates were then cleansed in acetone and methanol and treated with oxygen plasma. The substrates were subsequently coated with a polyimide alignment layer. The alignment layers were rubbed with a cloth, and the cell was constructed to ensure a planar alignment condition. The cell gap was controlled by mixing 15  $\mu\text{m}$  thick glass rod spacers into an optical adhesive. Samples were prepared by mixing 0.4–1.5 wt % of the photoinitiator Irgacure 369 (BASF), two right-handed chiral dopants (R1011 and R811, Merck), 6 wt % of an achiral LC monomer (RM82, Merck) or a right-handed chiral LC monomer (SL04151, AlphaMicron, Inc.), and a negative  $\Delta\epsilon$  (MLC-2079,  $\Delta\epsilon = -6.1$ ,  $\Delta n = 0.15$ ,  $T_{\text{NI}} = 102$  °C at  $\lambda = 589$  nm, Merck) or positive  $\Delta\epsilon$  (E7/ $T_{\text{NI}} = 58$  °C,  $\Delta\epsilon = 13.8$ ,  $\Delta n = 0.2253$  at  $\lambda = 589$  nm, Merck) nematic LC. The polymer stabilizing network was formed within the samples by photoinitiated polymerization with 100–250  $\text{mW cm}^{-2}$  of 365 nm light (Exfo) for 3–30 min. The photoinitiator concentration and the duration of UV exposure is the primary differentiator for the electro-optic responses reported here (red-shifting tuning, blue-shifting tuning, and bandwidth broadening). More specifically, for PSCLCs to exhibit bandwidth broadening, a small amount of I-369 (0.4 wt % or lower) is used to initiate polymerization, triggered by comparatively lower intensity light (here, 100  $\text{mW/cm}^2$  for 3 min). To induce red-shifting tuning, we formulated the PSCLCs with 0.5–0.8 wt % I-369 and initiated polymerization with 100  $\text{mW/cm}^2$  for 5 min. Finally, blue-shifting tuning was realized in PSCLC samples prepared with 1–1.5 wt % I-369 and initiated with comparatively high intensity exposure, 250  $\text{mW/cm}^2$  for 30 min. To ensure homogeneous curing conditions, the cell was rotated at an angular velocity of 2 Hz during polymerization. Although the samples prepared with more traditional, one-sided curing exhibit electro-optic responses similar to that reported here, the consistency and sample-to-sample repeatability are

improved by rotating the sample during curing. All materials were used as received without any purification.

**Experimental Setup and Measurements.** Transmission spectra were collected with a fiber optic spectrometer. Unless otherwise mentioned, the white light probe was right-handed circularly polarized. Transmission spectra were collected before, during, and after the application of dc fields with the scanning rate of 1  $\text{V s}^{-1}$  or directly applied to the target voltage.

## ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsomega.8b00453.

Summary of sample preparation conditions and control experiments comparing electro-optic responses of PSCLCs (PDF)

Control of color in PSCLC optical elements (AVI)

(AVI)

(AVI)

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### Notes

The authors declare no competing financial interest.

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