NI Light-Fueled Nanoscale Surface Waving in Chiral Liquid Crystal Networks

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ABSTRACT: Nano- and micro-actuating systems are promising for application in microfluidics, haptics, tunable optics, and soft robotics. Surfaces capable to change their topography at the nano- and microscale on demand would allow control over wettability, friction, and surface-driven particle motility. Here, we show that light-responsive cholesteric liquid crystal (LC) networks undergo a waving motion of their surface topography upon irradiation with light. These dynamic surfaces are fabricated with a maskless one-step procedure, relying on the liquid crystal alignment in periodic structures upon application of a weak electric field. The geometrical features of the surfaces are controlled by tuning the pitch of the liquid crystal. Pitch control by confinement allows engineering one-dimensional (1D) and two-dimensional (2D) structures that wave upon light exposure. This work demonstrates the potential that self-organizing systems might have for engineering dynamic materials, and harnessing the functionality of molecules to form dynamic surfaces, with nanoscale precision over their waving motion.

KEYWORDS: dynamic surface, artificial molecular switches, liquid crystal network, cholesteric liquid crystals, nanoscale motion

1. INTRODUCTION

Dynamic surfaces hold potential for engineering adhesion, optical and mechanical properties at the nanoscale, and therefore, they have a high impact in soft robotics, sensorics, haptics, etc.1 Remote spatiotemporal control over the response of dynamic surfaces would allow harnessing their full potential. Designing smart surfaces that respond to light would address this challenge and paves the way toward surfaces that perform work without producing waste and that can be locally activated over the desired time span. Remote dynamic behavior in smart materials is achieved when at least two main requirements are satisfied: (i) the material should contain a transducer of the external signal and (ii) it should possess machinery for enhancement of the signal to macroscopic scale by means of alteration of material size, shape, or other relevant parameters. Liquid crystal (LC) polymer networks are ideal candidates that fulfill these requirements when incorporating molecular photoswitches.2,3 Light responsiveness and electrosusceptibility allow preprogramming their optomechanical response and their nano- and mesoscale organization. Light responsiveness is achieved by the integration of photoswitches into the LC network. Photoswitches mediate control over the geometric parameters of the network often by light due to their conformational changes.4 Here, we control the network’s parameters using azobenzene photoswitches that mediate two main phenomena: (i) transduction of the molecular shape change of azobenzene into the polymer network and further up to the microscopic level, and (ii) reduction of the scalar order parameter of the LC network due to light-generated bend-shaped Z-form of azobenzene. Both phenomena usually work together and result in anisotropic deformation of the network.5 By adjusting the alignment of liquid crystalline molecules and their confinement, the light-driven deformation of the LC networks can be preprogrammed as bending, curling, and twisting of freestanding films6−9 and changing the surface corrugation of films when constrained on a solid substrate.10 There are several ways to create dynamic photocontrollable surfaces based on amorphous azobenzene-containing polymer systems11,12 and LC networks, which are mainly multistep and require lithography, masks, or exposure to interference patterns.13−21 However, dynamic surfaces can be fabricated by homogeneous photopolymerization of cholesteric layers with fingerprint texture.22−24 In our current work, we demonstrate how the unique property of chiral nematic liquid crystals to form highly ordered periodic patterns upon application of low voltage25 has been used for dynamic surface fabrication. The maskless and lithography-free approach proposed here (Figure 1a) allows for well-aligned (over the area of cm² scale) one-dimensional (1D) and two-dimensional (2D) periodic surface corrugations, which show reversible topography inversion under the action of light. The type of
surface profile relates to the type of electro-induced structure in the cholesteric organization and can vary from sinuslike to more complicated, e.g., sawlike. Moreover, this approach can be combined with mask exposure to reach spatially localized dynamic surface structures. Our findings demonstrate how control of liquid crystal alignment on a large scale can be expressed into surface profiles, which diversifies the toolbox of available methods for the fabrication of photo-motile surfaces and has potential application for sensors, haptics, and optics.

2. RESULTS AND DISCUSSION

2.1. Electro-Induced Periodic Patterns in LC Networks. Here, we have engineered light dynamic surfaces using the response of cholesteric liquid crystals to an electric field. When a threshold voltage $U_{TH}$ is applied to a planar cholesteric layer, in-plane modulated periodic structures can be observed in certain confinement conditions. When subjected to an electric field, cholesteric planes tilt sinusoidally with a defined period to minimize the free energy of the system caused by the hindrance of reorientation of the planes along the field, surface anchoring, and chiral torque. Spatial modulation of the orientation of LC molecules in these electro-induced structures and caused modulation of refractive index act as phase grating, which makes them appealing for practical applications as tunable diffraction gratings, beam steering devices, etc. Importantly, the emergence of a specific type of electro-induced structure is encoded by the helix confinement ratio ($d/P_o$, where $d$ is the thickness of the layer and $P_o$ is the pitch) as shown in Figure 1b.

The electro-induced structures of planar-aligned cholesteric layers can be roughly classified into one-dimensional (1D) and two-dimensional (2D). 1D structures include the so-called surface frustrated lying helix (SFLH) structure, where the axis of supramolecular helices lays in the layer plane and Helfrich-Hurault (HH) deformation refers to the periodic bending of cholesteric planes. SFLH and HH structures have a different mechanism of appearance and different optical properties. 1D periodic striped domain structures can be either parallel (HH-parallel and SFLH) or perpendicular (HH-perpendicular) to the rubbing direction of the polyimide aligning layer of electro-optical cell and are defined by the confinement of cholesteric helices. The second type refers to 2D (square) structures induced by the electric field in planar cholesteric layers with high confinement ($d/P_o > 2$). The intervals of supramolecular helix confinement, where a certain periodic structure exists, are revealed from the literature analysis and listed in Figure 1b.

Here, we succeeded in cross-polymerizing 1D and 2D electro-induced cholesteric structures formed in monomeric LC layers as is schematically shown in Figure 1a. The photopolymerizable monomeric mixture was composed of nematic mono- and diacrylates (C6BP, C6BPN, and RM257), azobenzene cross-linker (Azo-1) responsible for light-induced transformations, free LC molecules (E7) regulating the softness of the material, and a small amount of chiral dopant ((R)-BB) to adjust the cholesteric pitch (Figure 1c). After photopolymerization at the target applied voltage, and opening the cells, we have found that the top surfaces of the layers are periodically corrugated, reflecting the periodicity of the electro-induced structures. In contrast to previous reports where the free surface of cholesteric layers with homeotropic alignment (fingerprint texture) is corrugated due to Marangoni stress, in our case, the surface modulation is likely caused by the relaxation of internal anisotropic stresses built upon cross-polymerization and associated with the periodic structure of the LC director (see Figure S1). After removing the top glass substrate, the cholesteric material minimizes its free energy (compensates accumulated stresses) by periodic buckling of the free surface. The periodic surface corrugations are enabled by the free LC fraction in the materials that reduces the elastic modulus of the network facilitating its deformation. The material containing no free LC fraction E7 demonstrates a negligible (2–5 nm) surface corrugation (Figure S2). In the following sections, we discuss the surface topographies from all types of cholesteric structures in a line of growing confinement.
ratio from 1D to 2D with the focus on their light-fueled dynamic behavior.

2.2. Dynamics of 1D Periodic Surface Corrugations. A common feature of 1D HH patterns is the mechanism of appearance, which is similar to the development of photographs: the electro-induced transformation/bending of the initial planar cholesteric planes begins simultaneously and develops uniformly throughout the switched area of the layer. To create HH structures, we first used a monomeric mixture containing 0.1 wt % of (R)-BB and 8 μm thick cells. The resulting chiral confinement of 0.36 yields an HH-parallel structure. Figure 2a shows areas with stabilized HH-parallel structures obtained by local exposure of the cholesteric layer at 2.4 and 3.5 V. The rest of the sample was polymerized by a homogeneous exposure after switching off the voltage. It is seen that the contrast of the optical images is sufficiently enhanced by increasing the voltage and associated with the increase of the amplitude of the cholesteric plane deformation, which goes in line with previous experiments and simulations.25 Atomic force microscopy (AFM) study of these two areas revealed the sinuslike periodic surface corrugation where the depth increases from ∼60 to ∼100 nm with increase in voltage (see inset in Figure 2a).

The LC network is made photoresponsive by the incorporation of an azobenzene cross-linker in the design. When exposed to UV light, the surface becomes motile and starts a waving motion. The illumination inverts the surface topography, i.e., maxima (or surface hills) become minima (or surface valley) and vice versa, as is clearly shown in Figure 2b–d. In situ AFM study demonstrates the evolution of topography upon alternating irradiation with UV and visible light, which has a completely reversible character. Surface inversion upon UV exposure takes place fast with a frequency of about 0.5 Hz (see Figure S3), thanks to the efficient absorption of that wavelength (365 nm) by the azobenzene photoswitches. Further exposure of the surface to visible light leads to back inversion of topography passing through an almost flat state (Figure 2c) and recovery of the initial profile. Therefore, cycles of UV and visible light exposure generate wave-like dynamics of the surface at the nanoscale, as shown in Figure 2d,e. These waving dynamics are highly reproducible and can be performed many times without sign of fatigue (Figures S3 and S4).

The mechanism of surface topography inversion is caused by light-induced reduction of the order parameter due to populating nonmesogenic Z-state of azobenzene moieties upon UV exposure. The drop of order parameter generates activation forces leading to contraction of the network in a direction parallel to the LC alignment and expansion in opposite direction,38 which is expressed in surface morphing since lateral displacements are prohibited by the rigid substrate. Therefore, in the case of electro-induced cholesteric structures, which can be simply considered as orthogonal (in- and out-of-plane) periodic modulation of LC alignment, areas with opposite LC alignment work as to yield fast inversion of surface topography (see the scheme for SFLH structure on top of Figure 2b). Such waving behavior is mostly preprogrammed by the LC director configuration, which has been demonstrated by the targeted design of in-plane LC alignment...
yielding photoswitching between flat and corrugated surfaces; however, inversion of the surface profile has been observed in cholesteric elastomer layers upon heating−cooling cycles.

To fabricate dynamic surfaces from HH-perpendicular structures, we aimed at a confinement ratio of approx. 0.9, which has been achieved by doping a monomeric mixture with 0.25 wt % of (R)-BB. The HH-perpendicular structure was formed at 60 °C and 2 V applied to an 8 μm thick cell. After cross-polymerization and opening the cell, we have found a more complex surface profile than just sinusoidal, which mirrors a complex LC director field (Figure S1b). Small additional peaks (of 2−5 nm) on top and bottom of the corrugation can be distinguished (Figure 3a,b). UV exposure results in topography inversion with increased amplitude from ~40 to 100 nm (Figure 3b). The dynamics of topography displays a slow decrease of maximum and increase of minimum after switching off UV light, which indicates a negligible contribution of the photothermal effect (Figure 3c). Additional AFM study of the reference planar cholesteric surface, the surface without any electro-induced structures, has not revealed any dynamic behavior upon exposure. This fact together with a constant area under AFM profiles upon illumination (see Figures 3c and S5) indicates that light-driven inversion of topography proceeds with an insignificant change in volume, unlike previously reported dynamic surfaces based on cholesteric networks with fingerprint structures. In our case, operation of azobenzene cross-linker pulls the strands of polymer network along the alignment direction and reduces the order parameter due to nonmesogenic character of cis-Azo-1, which results in coherent force generation and therefore in the inversion of surface topography.

Surface topography can be controlled locally with precise spatial resolution. To demonstrate this, we inverted the initial surface profile of the HH-perpendicular structure by UV irradiation and then exposed it locally to a focused laser beam (532 nm) of ~40 μm in diameter; the surface topography was detected by AFM (Figure 4a,b). The maxima of the surface relief became minima in the locally exposed area, indicating inversion of topography. Surface profiles along lines B1 and B2 corresponding to the areas out and in the laser spot are shown in Figure 4b and confirm local surface morphing.

As we have shown above, the optical contrast and amplitude of modulation of HH structures can be enhanced by applying a higher voltage, which is limited by the voltage at which LC molecules become oriented perpendicularly to the plane of the layer (fully unwound state). It was shown that structures of HH-perpendicular type have quite a narrow voltage interval of existence. To achieve the highest amplitude of surface topography, we have used a monomeric mixture doped with 0.5 wt % of (R)-BB confined in an 8 μm cell gap (d/Po ∼ 1.78). To overcome the limitation mentioned above, we reduced the fabrication temperature to 55 °C that leads to a viscosity increase (consequently reducing the rotational

Figure 3. (a) Light-induced surface topography change of the HH-perpendicular structure (d/Po ∼ 0.89, cross-polymerization at 60 °C and 2 V). The inset on top shows the corresponding polarized optical image. (b) Surface profiles of the HH-perpendicular structure before and after exposure to UV light and (c) variation of the surface maximum and minimum as well as the reference surface (the surface without a structure) in the course of illuminations.
diffusion of LC molecules) allowing the use of higher voltages. The HH-perpendicular structure was cross-polymerized at 8.5 V. After opening the cells, we have found that the surface has a sawlike profile with quite sharp peaks (Figure 4c), which is in line with the LC director field in such structures at higher confinement (see also Figure S1c). The height of the relief is about 130 nm and reaches 200 nm upon UV light exposure. To demonstrate the nanoscale surface waving, the surface was continuously exposed to UV light while the green laser was switched on and off alternatively during in situ AFM measurements. The results are shown in Figure 4d. Time evolution of the topography (Figure 4e) displays repetitive surface profile inversion with negligible fatigue. It is important to note that the amplitude of the surface profile when green laser was on is smaller (~80 nm) in comparison with native amplitude, which is associated with the different photostationary states (Z-isomer rich) reached with simultaneous exposure to UV and green light.

The dynamic surfaces from the SFLH structure were successfully engineered as well from the monomeric mixture containing 0.5 wt % of (R)-BB filled into the cell of 6.3 μm thickness (d/Po ~ 1.4). They are characterized by sinuslike topography with relatively low amplitude (20–50 nm) and demonstrate similar light-induced inversion of surface profile (see Figures S6 and S1d). It is noteworthy that the thickness of the layer controls the periodicity of the structure but not the height (see Figure S7). The latter seems to be dependent on the type of electro-induced structure encoded and lies in the range from dozens to a few hundreds of nanometers.

2.3. Dynamics of 2D Surface Topography. As was shown in a number of works, 25,27,34,36 2D (square) structures may form under the voltage applied to the cholesteric layer if certain conditions are met: (i) a fairly high cholesteric helix confinement (d/Po > 2) and (ii) the absence of the defects in the cholesteric layer. The latter requirement is quite important since, at a given d/Po value, the formation of SFLH structures is facilitated by the defects presented in the layer. The voltage thresholds for the formation of SFLH structures are considerably lower than that for 2D ones, which makes the former dominant. In general, both structures coexist in dynamic equilibrium, which can be shifted by the regulation of the applied electric field. On the other hand, since we use a rather viscous monomeric melt, we are able to affect the equilibrium by changing the temperature. By lowering the temperature of the LC monomeric melt, the formation of the SFLH structure is significantly slowed down (or kinetically unfavorable) and the 2D structure starts dominating.

Therefore, a voltage of 6 V was applied to the cholesteric layer with a confinement ratio of 2.22 at 50 °C, resulting in the formation of a square 2D structure that was stabilized by photoinduced cross-polymerization. After opening the cell, the AFM study revealed the 2D periodic topography of the surface that mirrors the 2D electro-induced structure of the cholesteric layer (Figure 5a). Irradiation of the structured surface with UV light led to the formation of 2D relief but with a different arrangement of extremes. To understand how exactly the surface has been changed, we performed a threshold analysis of the AFM images obtained before and after exposure to UV light. Figure 5b shows the surface areas before irradiation with an amplitude of relief higher than 75 nm (in black) and the areas after irradiation with an amplitude of relief higher than 120 nm (in red). It is seen that the black and red areas alternate, indicating the inversion of the surface topography, which has been additionally confirmed by comparing the surface profiles before and after irradiation (Figure 5b).

Therefore, as far as we know, this is the first example of...
one-step production of the 2D periodic surface structure, which is able to reversibly invert a profile upon exposure to light.

3. CONCLUSIONS

We have developed dynamic surfaces that undergo a waving motion when fueled with light. The surfaces were fabricated using the following strategy: a weak electric field was applied to the chiral liquid crystal to develop 1D and 2D structures, and then, the structures were stabilized by cross-polymerization. The surfaces have a modulation depth in the range 20–200 nm, as engineered by the cholesteric pitch confinement and the applied voltage. In situ AFM studies have revealed that UV light exposure leads to an inversion of surface topography, which can be turned back by visible light exposure overall inducing a wavelike motion. These changes occur due to the pulling of polymer strands and the reduction of liquid crystalline order by bend-shaped Z-isomer of azobenzene fragments of the network. The configuration of liquid crystal arrangement in electro-induced patterns affects the character of surface topography leading to sinusoidal, sawlike, or multipeak profiles. Our findings demonstrate how intrinsic properties of the cholesteric phase and rational design of the confinement conditions can be used to harness molecular operation of azobenzene photoswitches towards motile surfaces. These layers with highly periodic and dynamic surface topography could be used for the manipulation of microscopic objects in haptics, and as tunable diffraction gratings in optics.

4. EXPERIMENTAL SECTION

4.1. Materials. We used a photopolymerizable cholesteric liquid crystal (Figure 1a), which is a mixture composed of LC monomers RM257 (30 wt %), C6BP (40 wt %), C6BPN (15 wt %), cross-linkable azobenzene switch Azo-1 (5 wt %), and 10 wt % of low molar mass liquid crystal E7 (Merck). All reactive monomers were purchased from Synthon Chemicals and used without additional purification. To induce cholesteric mesophase, a small amount (0.05–0.5 wt %) of bridged-binol ((R)-BBdopant was added to the monomer mixture as well as traces of photoinitiator Irgacure 819 (Ciba) to initiate photopolymerization. The helical twisting power of (R)-BB dopant was estimated in the used monomeric mixture (at 60 °C) as −44.7 μm−1 (wt %) using a wedge cell (E.H.C. Co. Ltd.).

4.2. Sample Preparation. To fabricate layers with periodic electro-induced structures, the cholesteric mixture containing a certain amount of (R)-BB was introduced to the planar electro-optical cell (E.H.C. Co. Ltd.) at 70 °C above the clearing point of the monomeric mixture. The sample was slowly cooled to 50–60 °C (heating stage, Instec), and AC voltage (1 kHz) was applied for 30 min. Then, while applying the voltage, cross-polymerization was initiated with blue light for 5 min (Blue LED, 455 nm, intensity ∼ 100 mW/cm2). For localized polymerization, the illumination was performed through a microscope objective. After irradiation, the voltage was turned off and the cell was annealed in the oven at 60 °C overnight. The typical appearance of the sample is shown in Figure S8. One substrate was carefully removed with a razor blade, yielding the LC network confined on one glass substrate. The type of generated electro-induced periodic pattern was dictated by confinement ratio. The ratio was adjusted by varying the cell thickness (6.3, 8, and 20 μm) and cholesteric pitch (4.5, 8.9, 22.4, and 44.7 μm), which in turn was determined by the amount of chiral dopant R-BB (0.5, 0.25, 0.1, and 0.05 wt %, respectively).
4.3. Measurements. Surface topography was characterized by atomic force microscopy (AFM) using an NTEGRA Spectra setup from NT-MDT integrated with an inverted microscope (Carl Zeiss) and green laser (Coherent). The images were recorded in the tapping mode. Dynamic AFM studies were performed by in situ UV or visible light exposure of the samples while scanning. As UV and visible light sources for dynamic experiments, 365 nm light-emitting diode (LED) (Thorlabs, intensity ∼18 mW/cm²) and Edmund MI-150 high-intensity illuminator equipped with a cutoff filter (λ > 420 nm) were used, respectively. Polarized optical microscope BX51 (Olympus) was used for optical characterization.

■ ASSOCIATED CONTENT

▲ Supporting Information
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsami.0c20006.

Simulated LC configuration of electro-induced patterns; surface structures with different periods; surface wave of HH-parallel and SFLH structures upon cyclic light exposure; and the photo of patterned LC network (PDF).

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Notes
The authors declare no competing financial interest.

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