1. Introduction

Shape-memory polymers (SMPs) are fascinating stimuli-responsive materials that exhibit irreversible shape-changes upon external stimuli such as light, temperature, or humidity.[1-2] SMPs have the ability to achieve and retain a temporal shape through the so-called “programming” to further recover their original shape upon application of a specific stimulus.[3,4] In particular, shape-memory photonic structures, such as photonic crystals and cholesteric liquid-crystalline (CLC) polymers, have received attention as they exhibit simultaneous shape and structural color changes upon temperature changes.[5-8]

Polymer coatings protect everyday objects from environmental influences. Coatings are also widely used for aesthetic purposes, for their reflective or antifouling properties or for adhesion control. Coatings can be fabricated from liquid paints using brushes or an aerosol by spray-painting. The color and surface topography determine the functional properties of the coating which are typically static. Currently, stimuli-responsive coatings exhibiting dynamic properties in response to external (environmental) stimuli are receiving much attention for various applications, such as self-cleaning, smart adhesives, optical sensors, and displays.[9] For these coatings, it is appealing to develop stimuli-responsive paints.[10] Among the existing stimuli-responsive paints, dye-based paints are the most common color-changing paints owing to the thermal or light responsivity of the dye.[11-15] In contrast, photonic paints, in principle, are able to generate both structural colors and topographical changes.[16-21] Such paints are useful to adjust surface wettability and adhesion and/or the reflective and aesthetic properties of a coating. Photonic paints usually require delicate processing to promote the formation of the photonic nanostructure,[21-23] and brush or spray-painting is not possible. Recently, structurally blue colored silica spheres have been reported as photonic pigments that can be mixed with a binder to form static photonic paints that can be brush-painted.[24] Nevertheless, it still remains a challenge to fabricate stimuli-responsive photonic multicolor paints exhibiting optical and topographical changes.

Recently, we reported on multicolored CLC polymer particles that can be used to fabricate static photonic coatings.[25] In this
work, we use the CLC particles to fabricate triple shape-memory photonic paints that can be brush-painted to form stimuli-responsive multicolor coatings which exhibit localized multiple optical and topographical changes. The CLC particles are used as photonic shape-memory pigments\cite{26–28} that are dispersed in a shape-memory binder\cite{29–32} to fabricate light and temperature responsive paints (Figure 1, step 1). The different glass transition temperatures ($T_g$) of both the particles and the binder enable the programming of two coexisting temporal optical and surface topographical states. The micrometer-sized photonic particles are compressed at high temperature ($T > T_{g,\text{particles}}$) and stabilized at intermediate temperature ($T_{g,\text{particles}} > T > T_{g,\text{binder}}$) leading to a structural-color change. Subsequently, the surface of the binder is embossed with a diffractive grating at intermediate temperature ($T_{g,\text{particles}} > T > T_{g,\text{binder}}$) and stabilized at room temperature to generate the surface topography. The permanent properties are recovered stepwise by increasing the temperature to change the surface topography and optical properties. The incorporation of a light-responsive dye into the particles polymer network allows for a high spatiotemporal control of sequential recovery by locally heating upon illumination with different intensities of light.

2. Results and Discussion

To prepare the light and temperature responsive shape memory photonic paints, we synthesized cholesteric polymer particle pigments exhibiting blue, green, and red structural colors, respectively, by suspension polymerization of a CLC mixture containing a light-responsive azobenzene dye\cite{33,34} (Figure S1, Supporting Information).\cite{25} Bisphenol-A based ethoxylate diacrylate (Figure S1, Supporting Information) was used as the binder because it has a high refractive index ($n \approx 1.53$) similar to that of the particles ($n \approx 1.6$) and a glass transition temperature ($T_g$) after polymerization around 38 °C which is lower than that of the particles ($T_{g,\text{particles}} \approx 67$ °C) and higher than room temperature (Figure S2, Supporting Information). Multicolor photonic paints were prepared by dispersing blue, green, or red CLC polymer particles in the binder (40% in weight of particles) and adding benzophenone (2% in weight) as photoinitiator. Dynamic mechanical analysis of a 70 μm thick photonic polymer film prepared through photopolymerization of the photonic liquid paint reveals two well-separated glass transition temperatures which correspond to $T_{g,\text{binder}}$ and $T_{g,\text{particles}}$ (Figure S2, Supporting Information).

To investigate the triple shape-memory of our photonic paints, we first prepared uniform photonic coatings on a black substrate (see in the Supporting Information) treated with an ethanol solution of benzophenone\cite{35} to enhance the adhesion. We used the green colored paint to form a uniform photonic coating of 70 μm. The resulting coating is flexible due to the low $T_g$ of the binder and exhibits a uniform green color that is angular independent to the naked eye (Figure 2a). The green structural color arises from the reflection of the photonic CLC particles as observed under the polarized optical microscope (POM, Figure 2b). The particles exhibit a green reflective centered dot which accentuates the angular-independent reflection properties that is attributed to the spherical confinement of the CLC layers.\cite{36} The scanning electron microscopy (SEM) image of the coating cross-section shows randomly embedded particles in the polymer binder (Figure 2c). Moreover, the cross-section
of a single particle (Figure S3, Supporting Information) reveals concentric arrangement of the cholesteric layers in agreement with the observed angular-independent reflection.

To create optical and topographical changes in the green colored coating, we first used a flat circular-mold ($\phi = 9.5 \text{ mm}$) made of stainless steel. After compression above $T_{g,\text{particles}}$ and cooling down to a temperature below $T_{g,\text{particles}}$ (Figure 3a, step 1), a blue colored circle was obtained. The SEM image of the cross-section reveals particles having an ellipsoidal shape with the long axis perpendicular to the compression direction suggesting uniform deformation of the micrometer-sized particles (Figure 3d). The deformation induces a shrinkage of the helical structure leading to a blueshift of the reflected color (Figure 3c). Considering that the compression step takes place within the elastic regime of the particles ($T > T_{g,\text{particles}}$), the deformation applied to the particles can be estimated by applying Hooke’s law, $\varepsilon = \frac{E}{E_0}$, where $E$ is the rubbery modulus of the particles determined at the compression temperature (Figure S2, 

Figure 2. a) Green reflective photonic coating prepared by using green CLC polymer particles dispersed in the binder, b) polarized optical microscopy (POM) image of the coating surface, and c) scanning electron microscopy (SEM) of the cross-section of the coating.

Figure 3. a) Two-step programming of a circular pattern on the photonic coating using first a flat mold and then a diffractive grating mold, b) image of the programmed photonic coating, c) POM image of the compressed, noncompressed border, d) SEM image of the cross-section of the coating after the first compression step, e) 3D image of the embossed topography with 2D graph of the cross-section, and f) UV–vis spectra of the coating before and after compression.
Supporting Information) and $\sigma$ is the stress applied determined from the pressure and area of the mold. The estimated deformation ($\varepsilon \approx 21\%$) correlates well with the observed blueshift ($\Delta\lambda = 60$ nm, Figure 3f) as the deformation of the helical structure by applying the equation, $\varepsilon = -\frac{2(\lambda_f - \lambda_0)}{\lambda_f} = 22.3\%$, where $\lambda_0$ and $\lambda_f$ are the reflection peaks before and after compression, respectively, and the factor two takes into account the spherical configuration of the helical structure (note that the blueshift is given by half of the sphere).

Next, a circular mold ($\phi = 9.5$ mm) having a diffractive grating (Figure S4, Supporting Information) was used to emboss the compressed circular blue surface with a diffractive topography (Figure 3a, step 2) at a temperature between $T_{g,\text{particles}}$ and $T_{g,\text{binder}}$. After cooling down to below $T_{g,\text{binder}}$ a well-defined blue circle exhibiting reflective patterns as a rainbow when observed at different angles of incident light was obtained (Figure 3b) indicating the formation of a relief surface structure. The embossed surface topography exhibits a regular grating of sharp and symmetric peaks with a spacing of 3.32 $\mu$m (2D plot) accordingly to the diffractive grating mold specifications (Figure 3e). However, a height comparison reveals a peak height difference of around 200 nm between the coating and the diffractive mold, suggesting that the mold did not penetrate completely into the surface of the binder (Figure S4, Supporting Information). It is worth noting that both the blue circle and rainbow effect coexist and are stable at room temperature due to the reduced mobility of the polymer chains below the glass transition temperature. Programmed samples can be stored with no visible changes.

The recovery of the original optical properties of the photonic coating was carried out stepwise by sequentially increasing the temperature to $T_{g,\text{binder}}$ and then to $T_{g,\text{particles}}$. During the first step, we observe that the rainbow effect vanishes and interferometer images show that the peaks of the regular grating become smaller and less sharp at 40 $^\circ$C to eventually disappear at 50 $^\circ$C within seconds (Figure 4a,b). Afterward, when the temperature reaches 60 $^\circ$C ($\approx T_{g,\text{particles}}$), the reflection peak starts to redshift from 470 nm (blue) to eventually the original 530 nm (green) wavelength at 75 $^\circ$C (Figure 4c), indicating the recovery of the original spherical shape of the photonic particles (see Video S1, Supporting Information).

The color changes can be tuned by using photonic particles with different structural colors. For example, a similar shift, from 568 nm (green) to 614 nm (red), is observed for the compressed coating containing the red photonic particles (Figure S5, Supporting Information). The temperature response of the coating is similar for the different photonic particles as the particles have the same thermal and structural properties.

To demonstrate light-induced localized recovery of a pattern coating, we fabricated a steering wheel mold ($\phi = 9.5$ mm) made of stainless steel to stamp a visual blue steering wheel pattern on a green photonic coating after compression above $T_{g,\text{particles}}$ and cooling down to a temperature between $T_{g,\text{particles}}$ and $T_{g,\text{binder}}$. Then, we used the circular diffractive mold (Figure S4, Supporting Information) on the steering wheel pattern to emboss the diffractive topography on the noncompressed areas in contact with the mold (the same programming conditions were used as detailed in Figure 3a). The programmed coating exhibits a well-defined blue pattern of the steering wheel that, when observed from different angles of incident light, reveals optical patterns as a rainbow (Figure 5, left image). We first removed the grating topography on half of the steering wheel by heating upon illumination with blue light (455 nm, 150 mW cm$^{-2}$) for 40 s. During light irradiation the

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Figure 4. a) Images of the thermally triggered recovery process, b) 3D images of the topography at different temperatures, and c) UV–vis spectra of the compressed area at different temperatures.
exposed area reaches around 50 °C (Figure 5, middle image) removing only the topography. Afterward, we erased different parts of the steering wheel pattern (Figure 5, right image) by illuminating with blue light (312 mW cm\(^{-2}\)), increasing the temperature of the coating up to \(\approx 80^\circ\text{C}\) (Video S2, Supporting Information).

To investigate the photothermal response of the light-responsive dye, we studied the temperature increase in a nonprogrammed photonic coating upon blue-light (455 nm) illumination at different light intensities (see the Supporting Information). From the experimental data (Figure S6d, Supporting Information), we confirm that the absorption of light by the azobenzene dye can effectively increase the temperature of the coating up to \(T_{g,binder}\) (150 mW cm\(^{-2}\)) or \(T_{g,particles}\) (312 mW cm\(^{-2}\)) to function as a trigger for the triple shape-memory. Moreover, the presence of the black substrate increases the maximum temperature achieved and moderates the temperature increase rate due to heat transport throughout the thickness (Figure S6b,c, Supporting Information).

Next, triple shape memory paints with different colors were used to create a multicolor painting. We brush-painted a multicolor flower by making use of a red-green-blue tricolor palette (Figure 6, step 1 and flower-making process in Figure S7a and Video S3, Supporting Information). Afterward, we programmed a green-to-blue colorshift by compressing the core of the flower at \(T > T_{g,particles}\) and cooling down to \(T_{g,particles} > T > T_{g,binder}\) (step 2.1). Then, we generated a surface topographical change around the core of the flower by embossing the circular diffractive mold and cooling down to \(T_{room}\) (step 2.2). The programmed photonic flower exhibits uniform blue-reflection from the core and reveals patterns as a rainbow upon illumination at different angles of incident light (Figure 6, right image). Recovery of the original optical state (steps 3.1 and 3.2) can be directly triggered by temperature increase or upon controlled illumination with blue light. We also designed an artistic painting of a landscape including shadows (Figure S8a, Supporting Information). In this painting, we applied two layers: in the first layer we painted the landscape by using the tricolor palette. In the second layer we used the carbon black (1% in weight) dispersion in the binder as black ink to make the tricolor palette. The triple shape-memory was programmed in two different areas of the landscape: first, the particles were compressed with a river-shaped mold to generate the illusion of water flowing. Then, a rainbow effect was created in between the mountains by changing the surface topography with the circular diffractive mold (Figure S8b, Supporting Information). The recovery process is shown in Video S4 in the Supporting Information. Painting and programming these stimuli-responsive photonic coatings can be also envisioned in a large-scale, high-speed roll-to-roll single process, for instance using flexographic printing and temperature controlled patterned metal rolls.

Initial experiments (Figure 7) also demonstrate the use of these paints to encode information by taking advantage of the circular polarized light properties of the photonic particles. We used green photonic paints having cholesteric particles reflecting left-handed and right-handed polarized light, respectively. The secret code “o c 3” was painted with right-handed paint and hidden in the shape of a fish painted with left-handed paint. When observed under normal light, the paint shows a drawing of a fish (left image). In contrast, when illuminated with right-handed circularly polarized light, the outline of the fish vanishes and the secret code is revealed (middle image).
Upon illumination with left-handed circularly polarized light, the outline of the fish is revealed (right image).

3. Conclusions

Triple shape-memory consisting of optical and topographical changes has been demonstrated in multicolor brush-painted photonic coatings. The photonic liquid paints have been prepared by dispersing cholesteric micrometer-sized polymer particles in a shape-memory binder and directly applied on a substrate to form dual-responsive photonic polymer coatings. The optical changes have been programmed in two-steps: first, the particles have been mold-compressed at high temperature to obtain a blueshifted pattern. Then, the surface topography has been embossed with a diffractive grating at intermediate temperature to generate a rainbow optical effect. Both optical and topographical changes have shown to be stable and coexist in the particles. Blue, green, and red photonic liquid paints have been used to brush-paint multicolor polarization dependent artistic paintings. These temperature and light responsive triple shape-memory photonic paints can be used to fabricate stimuli-responsive coatings and might find applications in the field of optical sensors, responsive decoration, and smart adhesives.

4. Experimental Section

Fabrication of the Photonic Paints and Coatings: First, the black substrate of the coating was prepared by forming a black layer of 500 μm thickness consisting on a dispersion of carbon black (1 wt%) in bisphenol-A ethoxylate (1.5 ET/phenol) diacrylate (Sigma-Aldrich) monomer and 1% in weight of photoinitiator Irgacure 891 (Chemische Industrie Basel Inc.) in a polyvinyl alcohol (PVA)-coated glass cell. The cell was exposed to UV light for 5 min each side to ensure full polymerization of the bisphenol-A monomer. Afterward, one glass plate was removed and the exposed surface was treated with a benzophenone solution in ethanol (3% in weight) by drop-casting and further spin-coating to remove the excess. The solvent was then evaporated at 40 °C for 15 min leaving the black substrate ready for paint application. The liquid, multicolor paints were prepared by dispersing blue, green, and red cholesteric particles, respectively, in the bisphenol-A monomer (40% in weight) and adding 2% in weight of benzophenone as photoinitiator.

For the preparation of photonic coatings, a thin 70 μm photonic layer of the liquid paint was formed on the black substrate by using a PVA-coated glass plate and spacers. The cell was exposed to UV light for 5 min to ensure completion of the polymerization of the bisphenol-A monomer and then opened by dissolving the PVA coated layers in hot water (60 °C) for 5 min.

For the fabrication of photonic paints, the liquid paints were brush-painted onto the black substrate and further polymerized with UV light for 5 min under N\textsubscript{2} atmosphere. Afterward, the surface of the paints was treated with the benzophenone solution to add an extra thin layer of bisphenol-A monomer (without particles) on top of the paint by using another PVA-coated glass plate (when uniform thickness was necessary) or directly brush-painted (as a varnish layer). The new layer was polymerized by exposing to UV light for 5 min. Finally, the cell was opened by dissolving the PVA coated layers in hot water.

Characterization Methods: Optical images of the photonic coatings were taken by POM using a microscope (Leica DM2700M) equipped with a Leica MC170 HD 370 high-resolution camera in reflection mode under crossed-polarizers. Cross-sections of the photonic coatings were analyzed by SEM using a JEOL SEM JSM-IT100: the samples were cut at room temperature using a razorblade, placed on sticky film and sputter-coated with a gold target at 60 mA over 30 s. The reflection of the photonic coatings was measured through UV–vis spectroscopy by using a PerkinElmer LAMBDA 750 with a 150 mm integrating sphere over a range of 250–1000 nm (wavelength) at different temperatures with a coupled heating stage. Thermomechanical properties of the binder, photonic film (particles dispersed in the binder), and cholesteric film (CLC mixture used for the particles) were measured by using a dynamic-mechanical-analyzer Q800 TA Instruments equipped with a tension-film geometry at constant oscillation frequency of 1 Hz and imposing a heating ramp of 3 °C min\textsuperscript{−1} from 20 up to 80–140 °C. The topography gratings were imaged and observed at different temperatures by noncontact optical interferometry using a Fogale Zoomsurf 3D optical profiling system with an external heating stage (Linkam).

Shape-Memory Experiments: The triple shape-memory programming was performed in different steps: first, the photonic paint was compressed under 20 Kg of pressure at 90 °C with a custom-made stainless steel mold by using a Tribotak (DACA Instruments) equipped with lower and upper heating stages. Then, the paint was naturally cooled down to 45 °C while maintaining the pressure applied. After thermal equilibration, the pressure was released and the paint was compressed again under 5 kg of pressure with a circular diffractive grating mold. After cooling down to room temperature, the pressure was released and the programmed paint removed from the equipment.

Thermally driven recovery experiments were performed on a heating stage (Linkam) with an external light source placed at different incident angles to generate optical effects with the topography. The recording of the recovery was processed by a Raspberry Pi Model 3B equipped with a Raspberry Pi camera module V2.1 extended with a Fujinon HF25SA-1 camera lens for manually adjustable focus.

Light-driven experiments were performed by using a Thorlabs light emitting diode lamp, emitting 455 nm blue light, with a collimation adapter to focalize the beam. The samples were placed at a distance of 10 cm from the collimator adapter. Blue light illumination on different areas of the sample was carried out in steps of 40 s (light ON) to achieve the plateau temperature and 1 min (light OFF) to allow the sample to cool down to room temperature. The recording of the recovery was processed with the camera setup used for the thermally driven experiments.
Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

cholesteric liquid-crystals, micrometer-sized particles, photonic materials, responsive coatings, shape-memory polymers

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