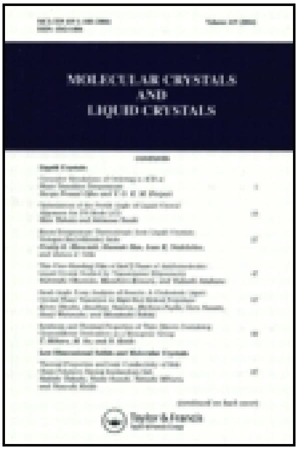

Molecular Crystals and Liquid Crystals

Three Techniques for Micropatterning Liquid Crystalline Polymers

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Version of record first published: 22 Sep 2010

To cite this article: Anastasia L. Elias, Michael J. Brett, Kenneth D. Harris, Cees W. M. Bastiaansen & Dirk J. Broer (2007): Three Techniques for Micropatterning Liquid Crystalline Polymers, Molecular Crystals and Liquid Crystals, 477:1, 137/[631]-151/[645]

To link to this article: http://dx.doi.org/10.1080/15421400701695687

Mol. Cryst. Liq. Cryst., Vol. 477, pp. 137/[631]–151/[645], 2007

ISSN: 1542-1406 print/1563-5287 online
DOI: 10.1080/15421400701695687

Three Techniques for Micropatterning Liquid Crystalline Polymers

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Liquid crystalline elastomers and polymers, which are able to undergo large, reversible deformations in response to an applied stimulus (including heat, pH, or electromagnetic radiation) could potentially be useful in a variety of microactuator devices. However, in order to compete with traditional silicon-based systems it is necessary to show that liquid crystalline polymers can be patterned with reduced dimensions without losing their functionality. In this article we investigate three different techniques which can be used to pattern liquid crystalline polymers on the micro- and nanoscale. These techniques are templating, photopatterning, and microtransfer printing. Advantages and disadvantages of each process are explored.

Keywords: artificial muscles; liquid crystalline polymers; microfabrication; polymer MEMS

Anastasia L. Elias and Michael J. Brett would like to acknowledge funding from the Natural Sciences and Engineering Research Council of Canada, the Alberta Ingenuity Fund, the informatics Circle of Research Excellence, and Micralyne Inc.

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INTRODUCTION

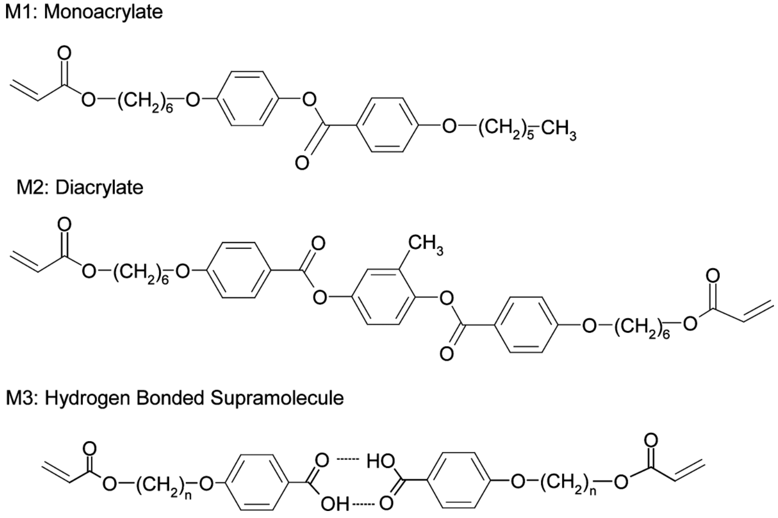
Liquid crystalline polymers and elastomers can be considered as artificial muscles, because they are able to transform various types of energy (chemical [1], thermal [2,3] and electromagnetic [4,5]) into mechanical energy [6]. These materials consist of anisotropic molecules (typically arranged in an ordered state) polymerized into a network with a flexible backbone. Liquid crystalline elastomers have been extensively investigated and described in the literature as they possess a variety of desirable characteristics which include: (1) their ability to undergo large deformations (up to a 3x change in dimension in one direction [7]), (2) the reversibility of these deformations, and (3) the fact that they can be made to deform in controlled directions by selective alignment of the anisotropic molecules from which they are comprised. For example, liquid crystalline polymer films that curl out of the plane have been demonstrated [8]. One potential use for these materials is as actuators in micro- and nanosystems [4,5]. Microfluidics and microoptics are just two examples of fields in which microactuators play an important role [9–11]. These fields have traditionally been dominated by silicon-based systems, which are fabricated using photolithography and traditional microelectronics processes. However, there is an emerging interest in replacing this stiff, costly material with low modulus materials which can undergo large deformations and can be patterned using simple, inexpensive, high-throughput techniques [12].

There are a limited number of techniques that can be found in the literature which describe micro- and nano-patterning of liquid crystalline polymers. One of us (D.B.) has shown that photopolymerizable liquid crystalline monomers can be irradiated in a mold with submicron features to produce a film with relief structures on the surface [13]. Van der Zande et al. have presented a process for fabricating liquid crystalline polymer optical retarder films for transflective displays [14]. These films consist of a checkerboard pattern (with dimensions ranging from 1 mm to 100 μm) of isotropic and nematic regions, which are made by photopolymerizing a reactive liquid crystalline monomer mixture through a mask in the nematic state, removing the mask, heating the unexposed material into the isotropic phase, and flood exposing the sample to preserve this phase. Sousa et al. have recently adapted this technique to make thermally-responsive actuators: with temperature variations the cholesteric region is able to undergo a larger strain than the isotropic area (~1% larger) [15]. Using soft-lithography and magnetic alignment, Buguin et al. have demonstrated a technique for making micron-scaled pillars of aligned nematic liquid single crystal elastomers [16]. UV-sensitive monomers are infiltrated into a PDMS master, aligned in a magnetic field, and photopolymerized. The pillars are then freed from the mould, which can be reused. Very good alignment can be achieved using this technique, and the pillars formed can also be actuated by heat.

While progress in producing aligned, patterned, liquid crystalline polymers and elastomers has been made, there is still a considerable amount of work that needs to be done before these materials can be conveniently integrated into micro-scaled devices. In this article we present and examine three different techniques for patterning photopolymerizable liquid crystalline polymers with features ranging from 400 μm to 100 nm. These techniques are: templating, photopatterning, and microtransfer printing. In templating, the structure of an inorganic, nanostructured thin film deposited by glancing angle deposition is replicated into a polymer film, which we will call the intermediate template. This intermediate template then acts as the master for the formation of the final liquid crystalline polymer helices with submicron dimensions. The photopatterning technique was adapted from that employed by Van der Zande et al. and Sousa et al.: a surface-aligned film in the cholesteric phase is photopolymerized through a mask. In our case, we do not heat the unexposed material to another phase and re-illuminate, but instead we remove the unreacted monomer in an appropriate solvent, leaving patterned lines of aligned liquid crystalline polymer. This allows a greater relative range of motion to be achieved when the structures are actuated, since for samples comprised of both cholesteric and isotropic areas the expansion of the cholesteric is effectively reduced by the fact that the isotropic region is also deforming (although to a lesser extent). The third technique that will be described is microtransfer printing. Stamps with 400 μm features are used to transfer liquid crystalline monomers onto glass substrates coated with alignment layers. The printed monomers are then photopolymerized in UV light, forming films approximately 3 μm to 4 μm thick. In this article each fabrication process will be described in detail, and the advantages and disadvantages of each will be discussed.

MATERIALS AND PROCESSING

Liquid crystalline polymers consist of anisotropic molecules which have been polymerized into a network. The mesogenic units employed throughout this article are shown in Figure 1. All three techniques presented are demonstrated using a mixture of M1 and M2. In this mixture the monoacrylate M1 is the primary constituent of the


M1: Monoacrylate

M2: Diacrylate

M3: Hydrogen Bonded Supramolecule

FIGURE 1 Liquid crystalline monomers. M1 and M2 may be used to form thermally-responsive polymers. M3 consists of 2 monoacrylic molecules, which are hydrogen bonded to form a supramolecule. This monomer can be combined with M2 to form polymers that can be sensitized to humidity by exposure to an alkaline solution.

material, and the diacrylate M2 acts as a crosslinker. These molecules are combined in a ratio of 22:3 (M1:M2 wt./wt.). This mixture is crystalline below 47°C, nematic from 47°C to 62°C, and isotropic above 62°C. To prepare a solid system, the molecules are mixed with a small amount ($<2\%$) of UV initiator (Irgacure184, CIBA), heated to the desired phase, and irradiated with UV light (in an oxygen-free environment). A chiral dopant (LC756, BASF) may also be added to achieve a cholesteric director profile.

For a uniaxially oriented thin film in which the molecules are aligned in one direction, the Young's modulus of the photopolymerized poly(M1/M2) material is 22 MPa perpendicular to the director, and 125 MPa parallel to the director. If the film is UV-illuminated while the monomer mixture is in the nematic phase, then the order of the phase is 'locked-in', even when the temperature is reduced. Furthermore, the order is preserved as the sample is heated towards a temperature of 215°C, around which a broad transition occurs to the isotropic phase.

In the microtransfer printing section of the article, another type of liquid crystalline polymer is also examined. As for the system described above, this material is prepared from three components: the principle reactive mesogen (M3, 86 wt.%), a reactive crosslinker (M2, 12 wt.%), and a photoinitiator (2 wt.%) [1]. Calamitic liquid crystallinity is observed in M3 under the action of hydrogen bonding. Reversible bonds are formed between adjacent carboxylic acid units, which create rod-like supramolecular structures with stable nematic phases [17,18]. Reactive groups are incorporated into every molecule, and therefore, these materials may be aligned and photopolymerized in the manner described above. For uniaxially oriented films the Young's modulus at room temperature is quite high: 3.9 GPa in the direction parallel to the long axes of the molecules, and 1.5 GPa in the perpendicular direction. Loss of order can be achieved by heating (as for the M1:M2 system), but may also be induced by exposure to alkaline solutions, generating a sensitivity to variations in pH and humidity as well as temperature.

TEMPLATING

In order to create high aspect ratio helical structures with submicron dimensions, a double templating process was employed [19,20]. This process is illustrated schematically in Figure 2, and described in greater detail below.

Master Fabrication

The master was fabricated with high control over structure, using glancing angle deposition [21–23]. In this process an inorganic source material (in this case SiO_2) is evaporated onto a substrate (in this case silicon), which is held at a high angle of incidence. In standard thin film evaporation processes the substrate normal forms an angle of $\sim 0^\circ$ relative to the flux from the vapor source, whereas here the substrate normal forms an angle of greater than 80° with the incident flux, as shown in Figure 3. In this configuration the thin film growth is dominated by shadowing and limited adatom diffusion, which results in the deposition of a highly porous thin film comprised of independent, columnar microstructures with high aspect ratios. The shape of these microstructures can be tailored by rotating the substrate during the deposition process. Different computer-controlled algorithms can be combined with information about the rate of deposition (as monitored by a quartz crystal thickness monitor) to engineer the exact shape of the structures. For the work described in this article, the substrate was rotated at a constant speed with respect to the rate of deposition, which results in the growth of helical structures, where the handedness is determined by the direction of rotation, and the pitch

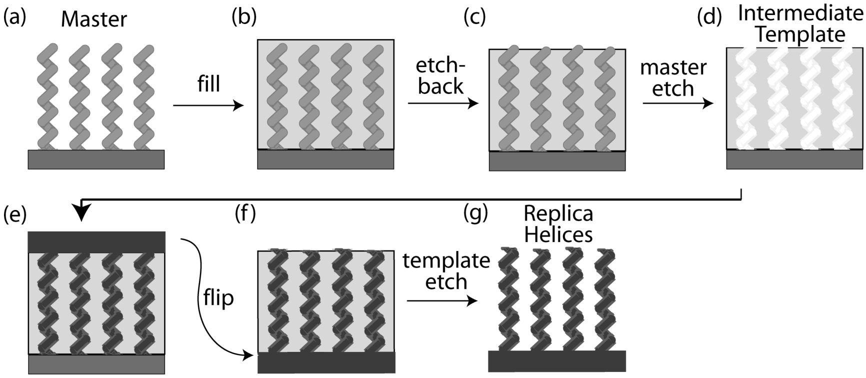


FIGURE 2 The double templating process. An inorganic master (a) is spin-coated with photoresist (b). This photoresist is UV-exposed and then etched slightly in basic developer in order to reveal the structures of the master film (c). The master film is removed, leaving the photoresist intermediate template (d). The intermediate template is filled with a liquid crystalline monomer mixture, which is then UV-polymerized (e). The photoresist/liquid crystalline polymer composite is removed from the original substrate by immersing the sample in liquid nitrogen (f). The photoresist template is dissolved in ethanol, leaving free-standing liquid crystalline polymer helices mounted on a substrate of the same material (g).

is determined by the speed. These master films are typically deposited over an entire 4 in. wafer at a time, allowing an extremely large number of helices to be fabricated in parallel. A detailed account of the glancing angle deposition process may be found in references [21–23].

Intermediate Template Fabrication

Once the master was deposited, its ‘positive’ structure was replicated into a photoresist ‘negative’. Photoresist was chosen as the material for the intermediate template as it is easy to process (using spin-coating), and has favorable etch-chemistry. To fabricate the intermediate template the master film was spin-coated with photoresist (HPR 504, Arch Chemicals). The resist formed a layer that just covered the top of the helical structures. The resist was then UV-exposed, and a small amount was removed from the top of the film in the appropriate developer (354, Arch Chemicals). Removing the top layer revealed the top of the helical structures, which allowed them to be fully removed by wet-etching in a buffered oxide etch (BOE, JT Baker). This left a continuous photoresist matrix with embedded helical pores,

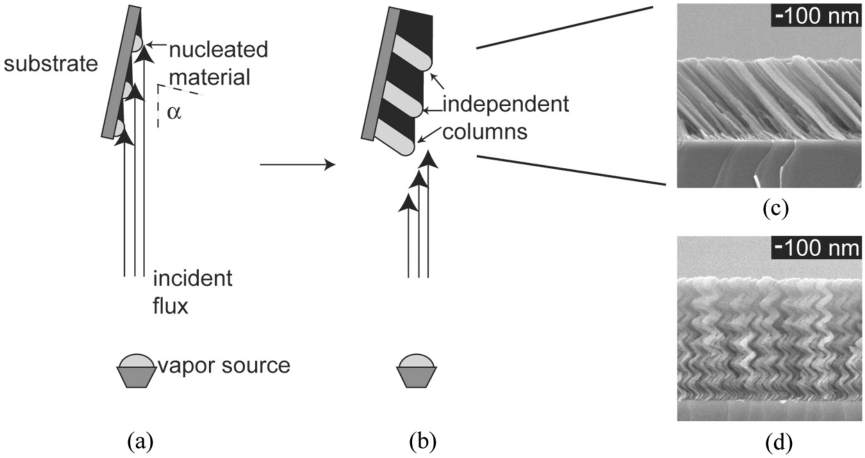


FIGURE 3 Glancing angle deposition. When the substrate is held at a high angle of incidence (α) with respect to the vapor source, parts of the substrate are shadowed from the vapor flux by nucleated material (a). If the diffusion of the adatoms arriving at the substrate is limited, the resulting film is highly porous and comprised of independent columnar structures (b). In the absence of substrate motion, these columnar structures resemble 'slanted posts', as shown in the scanning electron micrograph (c). If the substrate is rotated continuously during deposition, the resulting structures are helical (d).

which acted as the template for the fabrication of the final liquid crystalline polymer helices.

Liquid Crystalline Polymer Replica Helix Fabrication

Liquid crystalline polymer helices were made by heating the monomer mixture to a temperature just above the nematic-isotropic transition point of the mixture (63°C), spreading the mixture onto the intermediate template, and irradiating the sample with UV light in an oxygen-free environment. During this process a thick cap ($>15\,\mu\text{m}$) of liquid crystalline polymer formed on top of the intermediate template, which would become the substrate later in the process. The liquid crystalline polymer/template composite was then immersed in liquid nitrogen, such that thermal stresses forced the composite layer to separate from the substrate. The photoresist intermediate template was then dissolved in ethanol, leaving free-standing liquid crystalline polymer helices mounted on a substrate of liquid crystalline polymer (i.e., the thick cap), as show in Figure 4. In this case the alignment of the liquid

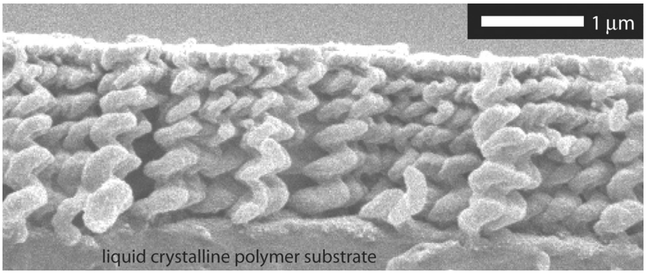


FIGURE 4 Liquid crystalline polymer helices. The liquid crystal polymer sample has been removed from the original silicon substrate, and the solid layer seen in the figure is composed of the same material as the helical structures.

crystalline helices was dictated by the surface of the helical pores. The director profile of these specific materials has not been measured, however the profile of a deuterated, low molecular weight liquid crystal infiltrated into the pores surrounding a helical master has been determined using nuclear magnetic resonance spectroscopy. In this experiment, it was found that the liquid crystals tended to align parallel to the surface of the helices at the same rise angle as the helical arms [24]. Therefore, in the case of the liquid crystalline polymer helices shown above, it is possible that the liquid crystalline moieties also follow the rise of the helical pores.

It was found that the director of the liquid crystal polymer network could be forced to assume a uniaxial configuration by modifying the fabrication process: after the liquid crystalline monomer was infiltrated into the template a transparent electrode (ITO) was placed on top of the polymer layer, as shown in Figure 5. An electric field was

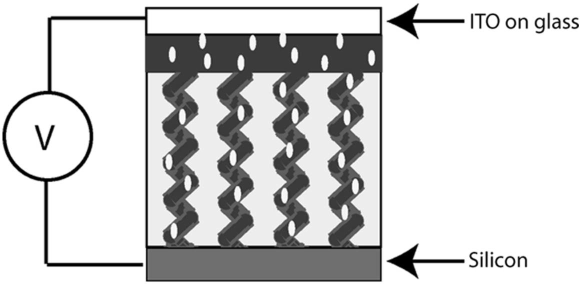


FIGURE 5 Electrical alignment of liquid crystalline monomers in the intermediate template.

then applied across the sample. Overall, the monomer mixture had a positive dielectric anisotropy, and the liquid crystalline monomers aligned parallel to the direction of the field. The sample was polymerized in UV light while the electric field was applied. The success of this technique could be measured optically to some extent (after the composite layer had been removed from the silicon substrate). The samples appeared dark when viewed directly between crossed polarizers, but began transmitting light when viewed off axis. As this is considered the optical signature of a homeotropically oriented birefringent film, we can conclude that the liquid crystalline moieties are generally homeotropically aligned.

Some advantages of the templating technique include the fact that a large number of structures can be fabricated in parallel over a large area, and that the characteristics of these structures (such as pitch, handedness, and number of turns) can be varied by altering the master template. It should also be possible to replicate some of the other complex three-dimensional structures achievable using glancing angle deposition with this technique (including zig–zags, vertical posts, and square-spirals), leading to a great deal of freedom to engineer the specific structures that are considered advantageous in particular applications. A disadvantage, however, is that the templates are destroyed during the fabrication process, which leads to additional processing steps as compared with a process in which the templates are reusable.

PHOTOPATTERNING

Photopatterning is a simple technique that can be used to pattern thin films of liquid crystalline polymers in two-dimensions, on substrates [14,15,25]. In this process an alignment layer is used to control the orientation of the liquid crystalline monomers prior to polymerization. Cells were constructed from glass slides coated with uniaxially rubbed polyimide (AL 3046, JSR), which were glued together with Norland UV adhesive. Spacers could be added to the adhesive to control the thickness of the cell gaps (typical spacer sizes were 10 μm and 18 μm). The liquid crystalline monomer mixture (a 22:3 wt./wt. ratio of M1:M2 with 2 wt. %initiator and 5 wt. %reactive chiral dopant added) was introduced into the cells in the isotropic phase by capillary force. Samples were then cooled to the cholesteric phase, and polymerized through a mask to preserve ordering in the exposed areas. Following polymerization, the cells were opened, and the unexposed material was washed away in acetone, leaving the final patterned structures

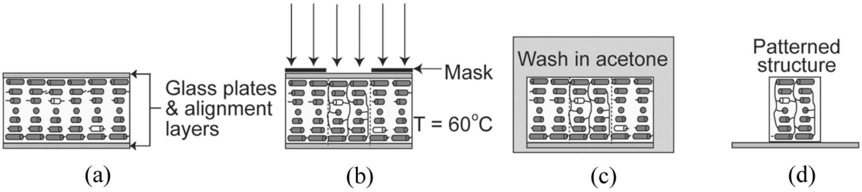


FIGURE 6 Process flow diagram for the photopatterning technique. A cell is filled with a mixture of monomers (a), heated into the cholesteric phase, and polymerized through a mask (b). The cell is then opened, and unexposed material is removed with acetone (c), leaving a patterned structure anchored on a substrate (d).

mounted on glass substrates. This process is illustrated schematically in Figure 6.

A sample patterned using this technique is shown in Figure 7. This sample was prepared using a mask with 100 μm lines with a 500 μm pitch. In this image the sample is viewed between crossed polarizers, which makes the non-liquid crystalline area between the cholesteric lines appear black.

For this work typical sample sizes were 1 in. x 1 in., although it is expected that the process can be scaled up to larger sample sizes provided that a collimated light source (a mask aligner, for example) is used. It is anticipated that much smaller structures will be achieved using appropriate masks (limited by the diffraction effects, which

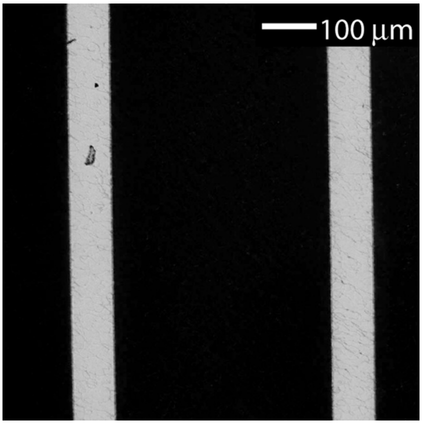


FIGURE 7 Patterned cholesteric lines. This sample is viewed between crossed polarizers in a microscope with a 10x objective.

may be enhanced by the thickness of the top plate of the cell). Although this photopatterning process is more material efficient than standard photolithography (*i.e.*, no dedicated resist layers are required because the active layer is patterned directly), one drawback of this process is that a considerable amount of material is still wasted during the acetone wash fabrication step. We attempt to address this issue using direct microtransfer printing.

MICROTRANSFER PRINTING

There are many advantages of the microtransfer printing process (shown in Figure 8) over other microfabrication techniques. From an environmental perspective, the printing process has little waste. The monomer mixture is transferred to the stamp, to the substrate, and then polymerized with minimal loss of material. Compare this process with techniques such as spin-coating or traditional lithographic processes, where excessive amounts of material are applied and removed in order to produce the desired structures. By comparison, printing leads to far less waste and also makes use of fewer organic solvents. Another advantage of printing is that it is easily scaled up in roll-to-roll processing, although for large-scale printing of liquid crystalline polymers temperature control of the stamps and substrates would be required. Nonetheless, this technique is promising for large-scale processing.

Preliminary investigations of printing of liquid crystalline polymers have been undertaken using commercial stamps. A 22:3 wt./wt. mixture of monomers M1:M2 containing 2 wt. % initiator and wt. % chiral

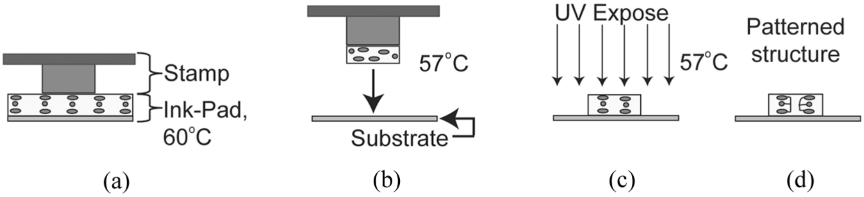


FIGURE 8 Microtransferprinting and photopolymerization of liquid crystalline monomers. In this process a stamp is pressed against an ink-pad which consists of the liquid crystalline material heated to within the cholesteric phase of the mixture (a). The stamp is retracted from the ink-pad and pressed against the substrate, which consists of rubbed polyimide coated on glass (b). The temperature of the substrate is also kept within the cholesteric phase. The printed material is polymerized in a flood exposure of UV light (c), which preserves the aligned, patterned structure (d).

dopant was spread onto a glass slide coated with uniaxially rubbed polyimide. This 'ink-pad' was heated to 60°C, and a commercial stamp depicting a monkey was pressed against it, allowing the monomer mixture to be transferred to the stamp. The stamp was pressed onto a glass slide coated with polyimide (which had been previously rubbed with a velvet cloth) and heated to 57°C. The pattern was polymerized in UV light for 5 minutes under a nitrogen environment. The resulting pattern is shown in Figure 9(a). By studying the appearance of the sample it can be seen that the liquid crystalline polymer is reasonably well aligned in the cholesteric phase, as the patterned material exhibits a red reflection band (appearing white in the grey scale image).

A mixture of 43:6:1 wt./wt./wt. mixture of M3:M2:initiator was also patterned, this time using a stamp depicting the date, as shown in Figures 9(b) and 9(c). Again the monomer mixture was transferred onto rubbed, polyimide-coated glass slides and photopolymerized. In the absence of a chiral dopant, uniaxial alignment of the liquid crystalline monomers was achieved, as can be seen in Figure 9. When the sample director is aligned at 45° between crossed polarizers, the printed material appears bright (Fig. 9(b)) due to the birefringence of the aligned material. When the sample is oriented with the polyimide rubbing direction aligned parallel to one of the transmission axes of the crossed polarizers, the printed material appears quite dark (Fig. 9(c)). Some smudging is visible in Figure 9(b), which may be avoided using a softer stamp (such as PDMS) and more controlled printing conditions (such as using an automated machine to apply and remove a specified amount of pressure).

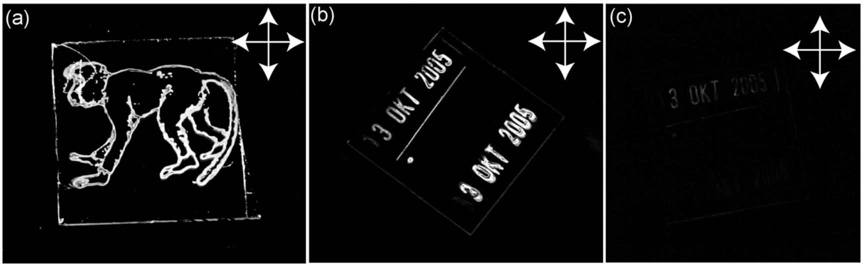


FIGURE 9 Microtransfer printed patterns. A monomer mixture containing M1/M2 and chiral dopant was printed and polymerized in the cholesteric phase (a). A monomer mixture containing M2/M3 was printed and polymerized in the nematic phase, and is shown oriented with the direction of alignment at 45° (b) and 0° (c) to crossed polarizers. The white arrows in each figure indicate the direction of orientation of the fast-axis of each polarizer. Note that each substrate measures approximately 1 in. by 1 in.

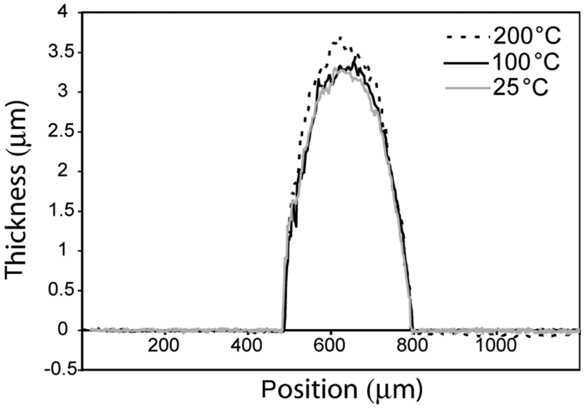


FIGURE 10 Thermal actuation of a microtransfer printed structure. The overall thickness of the structure decreases by 9% ± 2% (as a function of the height at room temperature) as the temperature is decreased from 200°C to 25°C.

In order to examine the thickness profile of the printed material and to demonstrate actuation of the structure, a cholesteric sample patterned using the monkey stamp was characterized using white-light interferometry. The profile of one section of the monkey's tail is shown in Figure 10, at three different temperatures. At 25°C the profile is rounded, with the greatest thickness occurring at the center of the printed structure. Note that in this figure the plane of the substrate is greatly compressed, as the scale of the x-axis of the graph spans more than 1000 μm while the maximum thickness of the structures is only 3.3 ± 0.05 μm. When the sample is heated the height of the structure increases, to a maximum of approximately 3.6 ± 0.05 μm. This corresponds to an overall change in height of 9% ± 2%. This change in height is completely reversible, and the curves shown were in fact measured as the sample was cooled from 200°C to 25°C.

Using commercial stamps we patterned liquid crystalline polymers with 350 μm feature sizes. This technique could potentially be ramped up to produce samples with high-throughput, while in future research we intend to concentrate on reducing the stamp dimensions and increasing resolution of the process by improving the stamp materials.

CONCLUSION

To incorporate liquid crystalline elastomers and polymers into microactuator devices, new techniques are required to pattern these materials on the micro- and nanoscale. The alignment of the liquid crystalline monomers is an important parameter that must be carefully controlled during the fabrication process, and in all cases techniques which produce substrate-anchored materials and avoid direct physical manipulation of the sample are greatly preferred. In this article three techniques for patterning liquid crystalline polymers have been presented: templating, photopatterning, and microtransfer-printing. These techniques are expected to be compatible with a wide variety of liquid crystalline monomers, and could potentially be used to pattern liquid crystalline polymers which respond to varying stimuli such as UV light, changes in pH, or variations in the electromagnetic fields surrounding the material. Assuming reliable techniques are found, these materials will one day find important uses in microactuator devices, lab-on-a-chip components, artificial muscle applications, and optical components.

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