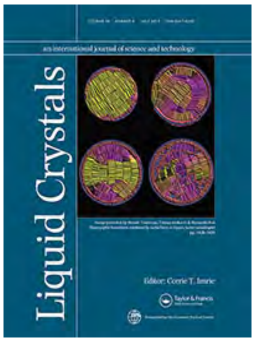

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High-diffraction-efficiency Fresnel lens based on annealed blue-phase liquid crystal–polymer composite

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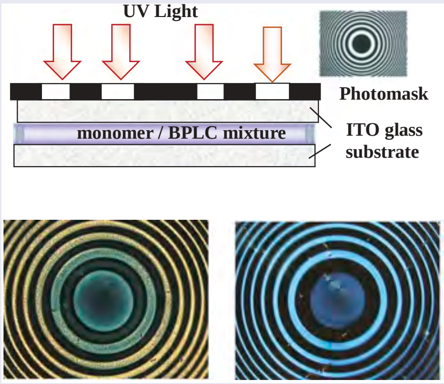
ABSTRACT
We demonstrated a relatively simple and effective method to fabricate a periodically isolated polymer wall of blue-phase liquid crystal Fresnel lens (BPLCFL) by employing a single-masking process of the ultraviolet (UV) irradiation, leading to excellent photopolymerisation-induced phase separation between blue -phase liquid crystal (BPLC) molecules and UV-curable monomers. Nevertheless, some uncured monomers would inherently reside in the BPLC-rich area and slightly inhibit the BPLC molecules realigned under the external electric field. To enhance the optical properties of the polymer-wall BPLCFL considerably, a novel technique for fabricating a pure BPLC zone is proposed that successfully expels the residual monomers from the BPLC volume using a thermal annealing process. Experimental results show that the maximum diffraction efficiency reaches ~36%, which approaches the theoretical limit of ~41%. Consequently, the annealing technique to purify phase-separated composite films has a strong potential to construct the BPLCFL in light of polarisation-free applications.



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Blue-phase liquid crystal; Fresnel lens; photopolymerisation-induced phase separation; thermal annealing; diffractive optics

1. Introduction

The liquid crystal Fresnel lens (LCFL) has been extensively adopted in various fields, such as with spectrometers [1], long-distance optical communication [2], projection displays [3], three-dimensional display systems [4] and variable optical data storage system using a zone plate modulator [5]. The pronounced electro-optic property and simple fabrication make it a very good candidate for electrically switchable lens devices. With the application of an electric voltage, the phase difference between the odd and even zones is induced by reorienting liquid crystal (LC) molecules. Thus, the optical diffraction efficiency of an LCFL can be electrically modulated. Compared to conventional processes likes electron-beam lithography techniques, the method used to fabricate electrically switchable LCFLs is quite simple, using polymer-stabilised LCs [6], dye-doped nematic LCs [7], polymer-separated composite film [8] and hybrid-aligned LCs [9], among other approaches. However, because of the intrinsic uniaxial anisotropy of LCs, the focusing properties of the LCFL rely strongly on the polarisation state of the incident light.

The polymer-stabilised blue-phase liquid crystal (PS-BPLC) has recently emerged as a promising candidate for photonics applications [10–16]. To overcome the polarisation dependence of the electrically switchable Fresnel lens, various techniques by using PS-BPLC have been proposed such as the periodic polymer slices structure, hybrid PS-BPLC grating, Fresnel even-zone electrode pattern, phase-separated composite film and so on [14–17]. For simplifying the fabrication process,

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we applied a unique method to realise the Fresnel binary zones by using photopolymerisation-induced phase separation (PIPS) [17–23]. The even and odd Fresnel zones are defined by a photomask, and a blue-phase liquid crystal (BPLC)/polymer binary Fresnel zone lens is then successfully generated by PIPS process. However, during the fabrication process, some residual monomers inherently reside in the BPLC-rich region, which slightly hinders the BPLC molecules from realigning under the external electric field and then degrades the electro-optic properties of the Fresnel lens. Besides, the fabricated BPLC lens could not avoid a significant amount of leakage light through the polymer boundary. For enhancing the blue-phase liquid crystal Fresnel lens (BPLCFL) performance, a critical technique is noticeably required to obtain a more complete separation of BPLC and polymer to improve the polymer wall structure and increase the purity of BPLC-rich zone without sacrificing the optical performance.

In this study to effectively purify the BPLC domain, a thermal annealing process is proposed that expels the remaining monomer from the odd regions and ameliorates the purity of the LC-rich regions. This purification method is similar to the principle of temperature-induced phase separation (TIPS) [23,24]. Based on the temperature difference, the phase separation of the BPLC and the photosensitive monomer is induced. When the thermal annealing treatment is thoughtfully controlled, more coalescence of BPLC droplets can be progressively obtained and make the remained monomers gradually expelled out of the BPLC domains. As a result, a perfect BPLC–polymer binary Fresnel zone with well phase separation is successfully achieved. According to the experimental results, the great success of the proposed TIPS annealing technique significantly improves the optical diffraction efficiency and response time of the BPLCFL. Therefore, we claim that the proposed annealing technique is extremely prospective for constructing the polymer–BPLC composite Fresnel lens device.

2. Experimental

2.1. Fabrication of polymer/BPLC composite Fresnel lens

To fabricate a switchable BPLCFL, the PIPS technique is applied, in which uniform binary layers of the BPLC and polymer are simply achieved by the spatially modulated ultraviolet (UV) light intensity of a photomask, as shown in Figure 1. The photomask with a circular pattern includes opaque odd zones and transparent even zones, in which the radius r_1 of the innermost zone designed is 0.4 mm and the radius of the nth zone (r_n) is given by $r_n^2 = nr_1^2$, with n denoting the zone number. The primary focal length f depends on the innermost radius r_1 as $f = r_1^2/\lambda$, where λ is the wavelength of the incident beam. Our Fresnel zone plate consists of 100 zones in an approximately 1 cm aperture and has a primary focal length f of ~25 cm for $\lambda = 632.8$ nm.

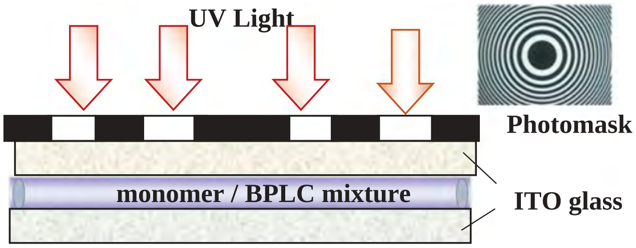


Figure 1. (Colour online) Schematic demonstration of the fabrication process of polymer/BPLC Fresnel lens.

To fabricate a BPLC Fresnel zone plate, we prepared a BPLC precursor comprising 57 wt% HTG135200-100 (an eutectic mixture of a rod-like nematic LC from HCCH, China), 3 wt% high twisting power and photo-stable chiral dopant R-5011 (HCCH) and 40 wt% photo-curable monomers [20% RM257 (Merck) and 20% TMPTA (1,1,1-trimethylolpropane triacrylate from Sigma Aldrich)]. The LC mixture has a positive dielectric anisotropy and, therefore, aligns parallel to an electric field. The physical properties of the nematic host are as follows: $\Delta n = 0.205$ at $\lambda = 632.8$ nm, $\Delta\varepsilon = 85$ at 1kHz and 21°C and clearing temperature is 90°C. The BPLC/monomers mixture was prepared uniformly by stirring. The BPLC precursor was subsequently injected into a cell containing indium-tin-oxide (ITO)-coated glass substrates by capillary flow at the isotropic temperature. The cell gap was fixed at approximately 15μm. After LC mixture injection, the sample in the isotropic phase was exposed to a beam of UV irradiation with a wavelength of 365 nm through the photomask. During the UV exposure, the monomer concentration was gradually consumed by the photopolymerisation and polymer, then aggregated in the unmasked areas and was separated from the BPLC because it does not dissolve in the solution due to its high molecular weight. Therefore, the polymer walls were formed in the irradiated even zone regions, and BPLC was segregated into the unexposed odd regions to realise an electro-optically responding Fresnel lens. The UV irradiation intensity highly influenced the polymerisation process and the rate of the phase separation, which are the most significant factors in determining the resultant structure of binary-phase lens systems. Here, the UV irradiation was controlled at a low intensity of 0.5 mW/cm^2 to form exceptional phase separation of the polymer and BPLC.

Nevertheless, the complete exclusion of the UV-curable monomer in the LC-rich domain after PIPS is difficult to achieve, so a second exposure of the UV light is required to remove the remaining UV-curable monomer. Thus, to photopolymerise the remaining monomers, the binary-phase BPLCFL sample after the PIPS process was cooled down to room temperature and then re-illuminated by uniform UV light with higher intensity without the photomask for 30 min to form the polymer network-stabilising BPLC layer in the odd rings in the previous work [17]. Although this second UV exposure solves the problem of the residual monomers, the excess dispersed polymer networks anchor the BPLC molecules and then degrade the electro-optic properties of the BPLCFL device. In addition, a considerable amount of light leakage occurs through the polymer network boundary. To overcome this serious problem, a technique to expel remaining monomers from the BPLC regions is imperative for developing the polarisation-free BPLC/polymer composite Fresnel lens. As the BPLC domain's high purity is successfully obtained, it can diminish the dispersed polymer networks in the BPLC domain and facilitate the higher quality of a polymer–BPLCFL composite, which is formed using spatially modulated UV intensity.

In this work, the thermal annealing process based on the principle of TIPS is proposed to squeeze the remaining monomers out of the BPLC-rich regions and considerably enhance the purity of the BPLC zones. Due to some residual monomers residing in the LC region after forming the polymer/BPLC composite layers, an annealing process is required to be done afterwards. The thermal annealing treatment is performed by heating the sample beyond the clear point to become isotropic and then slowly cooling it down at a decreasing rate of 0.2°C/min until room temperature. When temperatures drop lower than the clear point, the nucleation of BPLC domains is initiated and the BPLC domains start to grow into 'droplets'. Meanwhile, more growing LC droplets gradually coalesce together as temperatures decrease. Consequently, the remaining monomers are progressively driven towards the boundaries between polymer layers and LC regions, and then the high pureness of the LC domains can be successfully achieved. After the TIPS process, the UV illumination at an intensity of 3.0 $\mathrm{mW/cm^2}$ is sequentially performed to polymerise the well-expelled residual monomers near the BPLC/polymer boundaries.

2.2. *Characterising the voltage-dependent optic properties of BPLCFL*

The optical properties of BPLCFL mainly depend on the external electric field. In the absence of an external electric field, the BPLC in the odd zones is almost optically isotropic with an index $n_{\mathrm{iso}} = (2n_0 + n_\mathrm{e})/3$, in which n_o and n_e are the ordinary and extraordinary refraction indices of BPLCs, respectively. When applying the voltage on the BPLCFL, the director of LC molecules inclines to align parallel to the direction of the electric field, and the induced birefringence following the Kerr effect can be described as $\Delta n_{ind}(E) = \Delta n_s\left[1 - \exp(-(\frac{E}{E_s})^2\right]$ [25]. As a result, the electric field-induced phase difference between the odd and even zones of the BPLC composite Fresnel lens can be written as

$$\Gamma = \frac{2\pi}{\lambda}\left[n_0(E) - n_p\right]d \tag{1}$$

Here d is the cell gap, $n_o(E)$ is the induced ordinary index of BPLC, n_p is the refractive index of polymer wall and λ is the wavelength of the incident light. According to the Fresnel diffraction theory, the focusing diffraction efficiency of the Fresnel lens can be expressed as

$$\eta_m = \left[\frac{sin\left(\frac{\Gamma}{2}\right)}{\frac{m\pi}{2}}\right]^2 \tag{2}$$

where m represents the diffraction order. From Equation (2), when the phase difference of the proposed BPLCFL between the odd zones and the even zones is designed to be π, the maximum first-order $(m = \pm 1)$ diffraction efficiency of the Fresnel lens is theoretically ~41%.

Figure 2 shows the experimental setup for characterising the focusing properties of the BPLCFL, including the image quality, voltage-dependent diffraction efficiency and 3D spot intensity profiles. The He-Ne laser beam was magnified to approximately 1 cm just to cover the aperture of the lens size with an expander. A polariser was employed to change the polarisation angle of the incident light beam to determine the characterisation of the polarisationindependency of the proposed lens. Pinhole 2 was used to cover the light except for the first-order diffraction of the Fresnel lens. The light-focusing properties of the BPLCFL were measured using a charge-coupled device (CCD) and a photodiode detector, which were set at ~25 cm from the Fresnel lens.

3. Experimental results and discussion

The curing temperature in which the phase separation occurs plays a noteworthy role in determining BPLC–polymer composite structure and morphology [17,18]. The

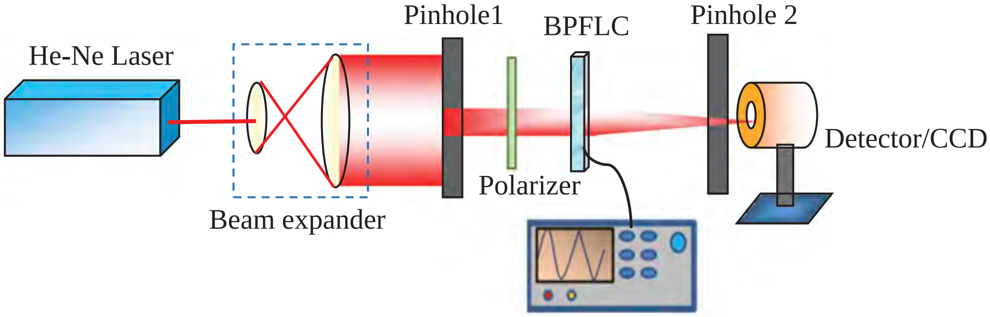


Figure 2. (Colour online) Experimental setup for measuring the focusing properties of the BPLCFL.

diffusion rates of BPLC and monomer molecules during the curing process strongly depend on the mixture's bulk viscosity. The decrease of the viscosity of the LC with increasing temperature facilitates the diffusion of the LC, the accumulation of the LC and then the growth of the domain size of LC. To study the influence of the curing temperature on the BPLCFL structure, two samples were cured at different temperatures. Figure 3 shows the formed BPLCFL structure cured at 50°C and 80°C. The dark areas indicate the polymer wall in the lens structure, whereas the coloured areas represent the BPLC domain. As the microscopic images in Figure 3(a) indicate, an incomplete phase separation between BPLC and polymer occurs due to slow diffusion rates of the BPLC mixture at a low curing temperature of 50°C. The BPLC molecules tend to get trapped in small areas in the polymer volume. Thus, BPLC molecules cannot efficiently separate from the monomers leading to the non-uniform polymer diffusion, as seen in Figure 3(a). This indicates that a slow diffusion rate of BPLC and monomers at low curing temperatures leads to the formation of the heterogeneous lens structures and that a certain concentration of LC micro-droplets is trapped in the polymer volume. Figure 3(b) demonstrates that BPLC molecules can efficiently separate from the polymer, as the temperature is increased to 80°C. As a result, the curing temperature during UV irradiation of the lens sample should be carefully controlled to achieve a good phase separation between BPLC and the UV-curable monomer efficiently.

Figure 4 shows the polarised optical microscopic photograph of the Fresnel BPLC zone plate at V = 0, 50, 100 and 200 V_{rms} with a frequency of 1 kHz. With no voltage applied, the sample exhibits the BPLC/polymer composite Fresnel zone structure. The odd and even zones show different colours, which means a complete phase separation of the BPLC and polymer was successfully obtained. When the applied voltage exceeds a threshold voltage (~10 V_{rms}), the colour in the BPLC odd zones started changing, as shown in Figure 4(b–d). Meanwhile, the appearance in the even region remained unchanged, which means no BPLC resided in those areas. When an external voltage was applied, the electric field-induced Kerr effect started to change the refractive index of the BPLC domain, whereas the refractive index of the polymer region remained unchanged. Hence, the phase difference between these two regions and the optical diffraction efficiency were changed gradually by increasing the electric field. As a result, the colour change in the BPLC odd zones indicates the electric field-induced lattice distortion, which is known as the electrostriction effect [25].

Figure 5 illustrates the focusing properties of the BPLCFL captured by a CCD camera. As the lens

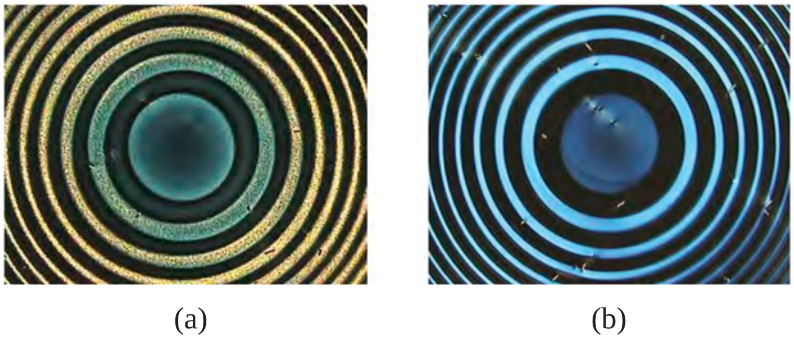

(a) (b)

Figure 3. (Colour online) The microscopic images of the sample prepared at curing temperatures of (a) 50°C and (b) 80°C under the cross-polarised optical microscope.

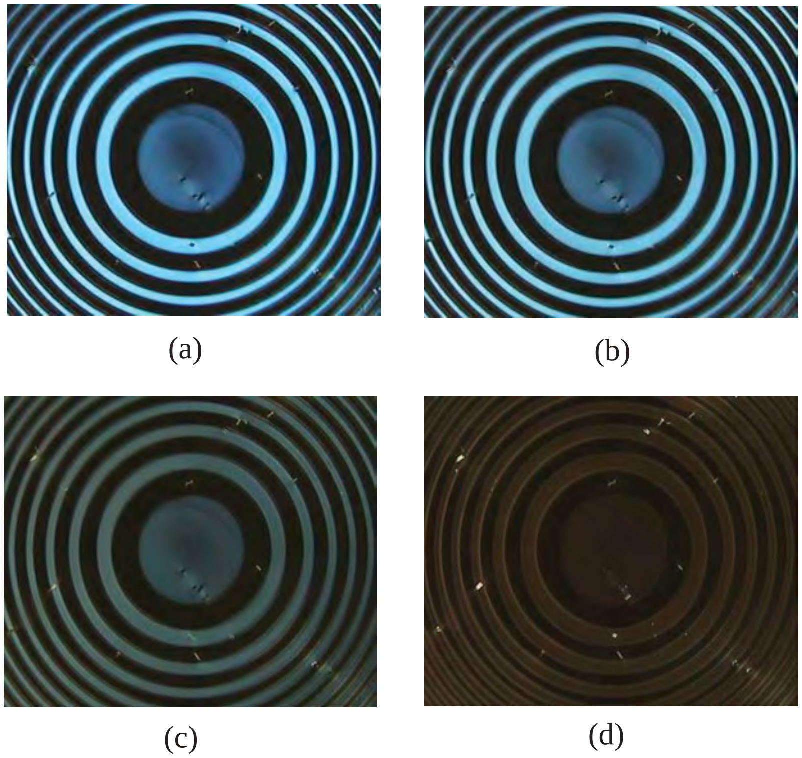


Figure 4. (Colour online) Microscopic images of the Fresnel BPLC sample at (a) V = 0, (b) 50 V_{rms}, (c) 100 V_{rms} and (d) 200 V_{rms} observed under crossed polarisers, respectively.

sample is absent, no focusing effect occurs, as shown in Figure 5(a). Once the BPLC Fresnel lens was present with no applied voltage, an apparent but smaller light spot is observed, as shown in Figure 5(b). As the applied voltage is beyond 50 V_{rms}, the focusing effect is enhanced, as shown in Figure 5(c,d). When the applied voltage is increased to about 180 V_{rms}, the focusing ability of the BPLC lens was found to approach the maximum. Based on the experimental results, the sample definitely behaves like a switchable lens.

The 3D intensity distribution at the primary focal point of BPLCFL with different voltages was also measured using a digital CCD camera. When the BPLCFL is in position, a sharp focus occurs at the primary focal point (~25 cm), as shown in Figure 6. In the voltage-off state, a slight focusing effect of the sample is observed. As the driving voltage increases, the peak intensity gradually increases, approaching the maximum at V_{rms} = 200 V.

To further assess the image quality of the proposed BPLCFL, a black piece of cardboard with a transparent number '7' was placed in front of the sample. Figure 7 demonstrates the recorded images of the '7' pattern as the CCD camera is located at different positions from the BPLC lens operated at V = 200 V_{rms}. When the CCD camera was placed at a distance of 20 cm from the BPLC plate (5 cm in front of the focal plane), two images were observed simultaneously, as shown in Figure 7(b). The bigger '7' image signifies the projected images of the '7' pattern without being diffracted, and the smaller '7' image corresponds to the first focus

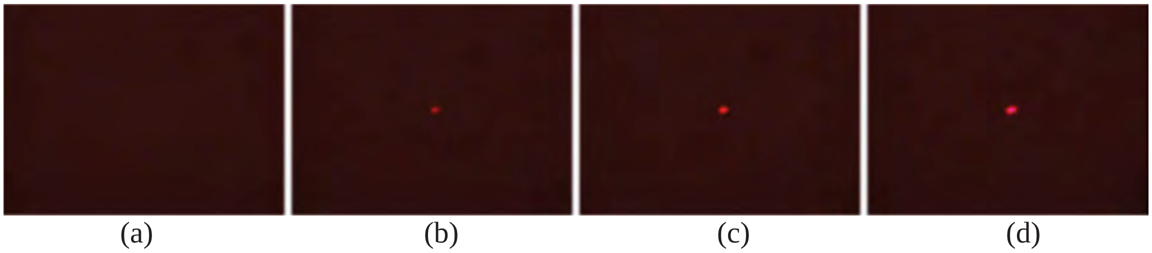
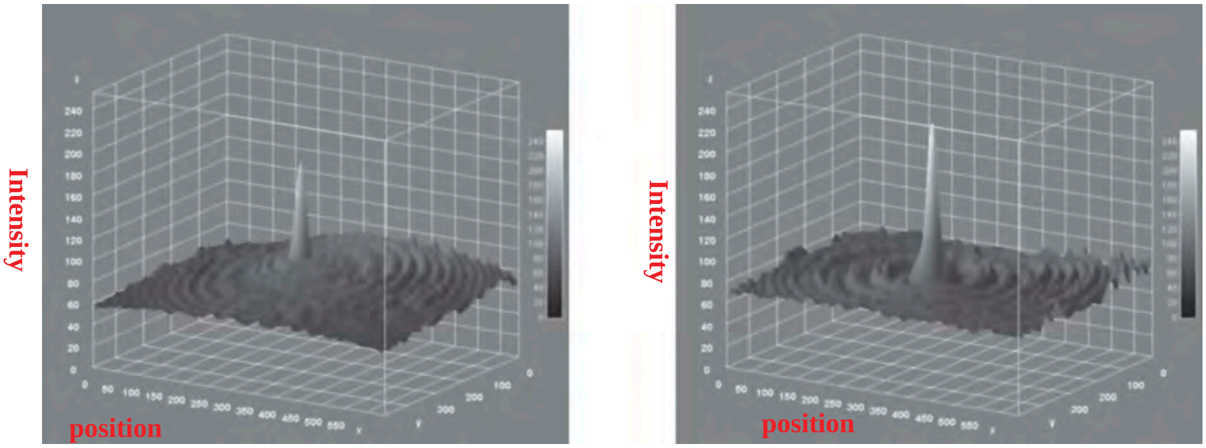
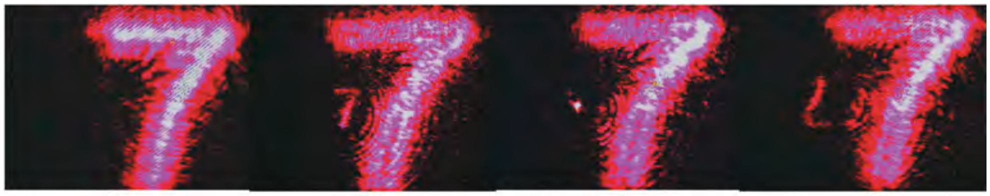


Figure 5. (Colour online) The observed laser beam images (a) without LC sample and with the sample at the applied voltage (b) 0 V_{rms}, (c) 50 V_{rms} and (d) 200 V_{rms}.



(a) (b)

Figure 6. (Colour online) Spot intensity profiles measured by the CCD camera under different conditions: (a) with the sample at V = 0 and (b) V = 200 V_{rms}.



(a) (b) (c) (d)

Figure 7. (Colour online) Diffracted '7' patterns (a) without LC sample and with sample recorded at different positions: (b) in front of the focal point, (c) focal point and (d) behind the focal point.

order. When the CCD was just in a focal point position (at 25 cm), the beam was focused to a bright spot, as shown in Figure 7(c). When the distance of CCD from the LC sample was changed to 30 cm, the smaller '7' image is reversed, as illustrated in Figure 7(d). According to the observed results, the proposed BPLC Fresnel zone plate can realise proper focusing and imaging performance.

The voltage-dependent diffraction efficiency of the proposed BPLCFL was also measured under different polarisation angles of the incident linearly polarised light, as shown in Figure 8. The diffraction efficiency is defined as $\eta = (I - I_o)/I_t$, here I denotes the transmitted light intensity at the primary focal point, I_o is the background noise and I_t is the total incident light intensity after passing through the sample. The experimental results demonstrate that the initial diffraction efficiency of the BPLC lens at the voltage off state is ~10%, which is due to the effective refractive index mismatch between the even and odd zones of the BPLCFL. When a voltage beyond the critical value of approximately 10 V_{rms} was applied, the LC directors in the odd rings begin to realign in the electric field direction, and the effective refractive index is then induced by the Kerr effect. Alternatively, the polymer in the even rings remained basically unaffected. When the external voltage increases, the phase difference between these two neighbouring zones increases, as

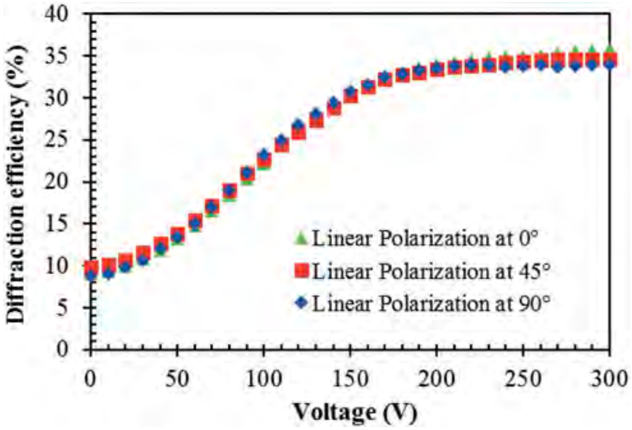


Figure 8. (Colour online) The voltage-dependent optical diffraction efficiency of BPLCFL.

does the optical diffraction efficiency. As the applied voltage increases to 200 V_{rms}, the diffraction efficiency nearly reaches the maximum value of 35.8%, which is not so far the theoretical limit. Because the reflection at two interfaces of glass–air happens as ~4%, the slightly lower optical diffraction efficiency of the proposed BPLCFL is mainly induced by the reflection at the two substrate–air interfaces and weak light scattering occurring at the interfaces between polymers and BPLCs. The high operation voltage of 200 V could cause electrode breakdown and severe hysteresis effect [26]. To lower operation voltage and enhance the performance of BPLCFL, better selection of polymer composition and proper control of the PIPS condition are critically required such as curing UV intensity and curing temperature and time, respectively.

The influence of the annealing treatment on the electro-optic behaviours of BPLC lens is also evaluated. Table 1 provides a summary of the maximum diffraction efficiency η_{max} and response time at applied voltage 200 V_{rms} of the samples with/without thermal annealing treatment, respectively. We observed that the annealed BPLCFL owns higher diffraction efficiency and shorter response time than that without thermal treatment. The electro-optic response of the proposed BPLC lens is appreciably improved by the thermal annealing process. Because a reduced amount of the dispersed polymer network existed in the BPLC region to anchor the reorientation of the LC molecules, the EO performance of an annealed BPLCFL can be notably ameliorated by the proposed TIPS process to successfully purify the BPLC domain. Therefore, the annealing process plays a critical role in optimising the formation of the polymer–BPLC composite system. In addition, because residual monomers can be effectively diminished by the thermal annealing process, the polymer zones can be initially realised by higher UV intensity to shorten the process time of PIPC; annealing treatment is then sequentially applied to effectively squeeze the remaining monomer out of the LC domain, which can effectively improve the electro-optic behaviours of a binary BPLC lens. As a result, the proposed annealing process can effectively improve the electro-optic behaviours of a BPLCFL and will be especially useful for the development of BPLC composite devices in the future. Moreover, we found the magnitude of the response time under a high electric field is several milliseconds, and this is because a strong electric field speeds up the electrostriction effect which attributes the response time much longer than that induced by the Kerr effect [27]. Because the value of response time induced by the electrostriction effect depends on the monomer concentration, optimum monomer concentration to form more stable polymer network in BPLC-rich domain is notably required to suppress the electrostriction effect, which takes less time for the electric field to finish the BPLC lattice stretching process. Therefore, a delicate balance between response time, hysteresis and operation voltage should be also taken into consideration to realise the high performance of PCLCFL device.

Table 1. Comparisons of the electro-optic properties of BPLCFLs with/without the annealing treatment, in which S1-A and S2-NA indicate the samples with and without the annealing treatment, respectively.

Properties sample	η_{max} (%)	T_{rise} (ms) @ 200V_{rms}	T_{fall} (ms) @ 200V_{rm}
S1-A	35.8	4	7.4
S2-NA	32.0	5.2	9.2

4. Conclusions

We have demonstrated an effective method for improving the performance of the electrically switchable BPLC composite Fresnel lens based on thermal-induced phase separation. The proposed thermal annealing technique successfully refines the BPLC-rich regions and considerably overcomes the problem of the residual monomer inherently residing in the LC region. As a result, the proposed annealing technique expedites the purity of the BPLC binary-phase lens containing a polymer wall structure and then optimises the electro-optical properties of the BPLC lens by lessening the dispersed polymer network in the BPLC-rich domain. Moreover, the focusing behaviour of the BPLCFL can be continuously controlled by a uniform voltage, and the maximum diffraction efficiency reaches ~36% regardless of the polarisation state of the incident light. With the merits of simple fabrication, polarisation independency and continuous modulation, the proposed BPLC Fresnel lens can have broad potential applications.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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