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Alignment of polymer network-stabilized ferroelectric liquid crystal using a magnetic field

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Polymer network-stabilized ferroelectric liquid crystals with homogeneous alignment have been produced in cells without a surface alignment layer. In this technique, a crosslinkable monomer is mixed into a ferroelectric liquid crystal and polymerized in a magnetic field to form a polymer network that will stabilize the alignment of the ferroelectric liquid crystal. The concentration of the monomer is an important factor in achieving alignment of the ferroelectric liquid crystal. Both the morphology of the final composite layer and the molecular alignment of the host FLC are affected by the curing temperature at which the UV curing of the sample is started.

1. Introduction

There is great interest in producing fast switching polymer network stabilized liquid crystals using ferroelectric liquid crystals [1–3]. Polymer network-stabilized ferroelectric liquid crystals (PNSFLCs) can be produced by mixing a ferroelectric liquid crystal with a small amount of crosslinkable monomer followed by polymerization of the monomer to form a solid network in the liquid crystal. PNSFLCs have attracted considerable attention for both their scientific interest and their potential applications in display devices. They possess very good electro-optic characteristics such as fast switching and optical bistability [4], and are promising materials for display applications. On the other hand, the formation of a polymer network in a liquid crystal environment will promote certain features of the composite.

One of the most interesting effects of polymer network-stabilized liquid crystals is that of the polymer network on the molecular alignment of the host liquid crystal. Studies carried out on nematic-based polymer network systems have revealed that a polymer network formed by *in situ* polymerization of a crosslinkable monomer in an aligned liquid crystal is oriented and anisotropic, and can stabilize the alignment state of the host liquid crystal [5–7]. However if the polymerization of the monomer was carried out when the liquid crystal was in the isotropic state, the polymer network formed could destroy the alignment of the liquid crystal [6]. This suggests that a prerequisite for obtaining an aligned LC/polymer composite layer is to produce an aligned liquid crystal environment prior to the polymerization of the monomer. In most current studies, an aligned liquid crystal environment is promoted using conventional surface alignment techniques, in which the inner surfaces of the substrates are coated with a thin layer of polymer and then rubbed, or an obliquely deposited SiO layer. In this technique, the alignment layer is not removed from the substrates, and acts together with the polymer network to align the liquid crystal in the cell. However, in PNSLCs the use of the alignment layer for the alignment of the liquid crystal is not necessary since the polymer network can impose strong anchoring on the surrounding liquid crystal molecules and maintain the alignment state of the liquid crystal. Methods for obtaining aligned PNFLCs in alignment layer-free cells, such as applying a mechanical shear during the polymerization of the polymer precursor [8] and the use of a linearly polarized light beam [9], have been demonstrated.

Recently, we have reported that homogeneously aligned polymer-stabilized ferroelectric liquid crystal layers in alignment layer-free ITO glass cells can be produced using a magnetic field [3, 4]. In this technique, a small amount of crosslinkable monomer was mixed into a low molar mass ferroelectric liquid crystal. A

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magnetic field was applied to the mixture to promote an oriented environment. The monomer was then polymerized and formed a solid polymer network which, in turn, froze the alignment state of the host liquid crystal. In this technique, the surface alignment layer is omitted, and the surface treatment for the substrates is simplified.

In this paper the use of an external magnetic field in the molecular alignment of PNSFLCs is presented. The influence of the concentration of polymer and the effects of sample temperature during polymerization on molecular alignment of the host liquid crystal are demonstrated.

2. Experimental

In this investigation a UV curable optical adhesive, NOA65 (Norland, USA), was used as prepolymer. The liquid crystal used was a commercial ferroelectric mixture SCE3, Merck (UK) Ltd, which has the phase sequence:

$$\mathrm{Cr}(<0)\ \mathrm{SmC}^*\ 74\ \mathrm{SmA}\ 105\ \mathrm{N}^*\ 136\ \mathrm{I}.$$

The liquid crystal was mixed with a small amount, <10 wt %, of NOA65. The mixture was mechanically stirred at an elevated temperature (~10°C above the clearing point of the host liquid crystal) to achieve uniformity. The phase transition behaviour was examined using a polarizing microscope. The sample cell was constructed using two ITO coated glass plates without any surface treatment other than a cleaning operation. Two opposite edges of the cell were sealed using epoxy glue, Quick Set Epoxy Adhesive (RS 554-850); the other two edges were left open. The thickness of the cell was confined to 20 μm using glass bead spacers. The empty cell was heated to an elevated temperature. A small amount of SCE3/NOA65 mixture was put at one of the openings of the hot cell, and forced into the cell by capillary action. An external stabilized magnetic field of 4 kG was applied parallel to the plates of the cell while the sample was at the high temperature. Then the sample was carefully cooled in the presence of the magnetic field. In the present studies, the samples were cooled to room temperature at a rate of about 1°C min^{-1}. The temperature of the sample was controlled by a Linkam TMS90 temperature system (Linkam Scientific Instruments Ltd, England). A Scientifica & Cook electrical magnet system (Scientifica & Cook Electronics Ltd, England) was used to produce the magnetic field. The polymerization of the mixture was carried out by UV light, also curing the adhesive, during the cooling process and in the presence of the magnetic field. A high pressure mercury lamp (Philips 93136E), with a wavelength of 365 nm, was used as UV light source. The intensity of the UV light was 9 mW cm^{-2} on the surface of the substrate. The UV curing lasted for about 60 min. A long duration curing was necessary to get a highly complete polymerization of the mixture with a low remaining concentration of monomer [5].

The alignment of the PNSFLCs was examined by means of optical microscopy. For a layer of FLC, when all molecules in the layer are homogeneously aligned and orient in a unique direction, the FLC layer can be assumed to be uniaxial [10, 11], and it acts as a birefringent wave plate. When the aligned FLC layer is put between two crossed polarizers, the optical transmission of the system for a monochromic light beam can be given as [12]

$$I(\Phi) = I_0 \sin^2(2\Phi) \sin^2(\pi \Delta n d/\lambda) \tag{1}$$

where I_o is intensity of the incident light, Φ is the azimuthal angle of the molecular director relative to the polarization direction of the polariser, Δn is the birefringence of the liquid crystal, λ is the wavelength of the incident light, and d the thickness of the FLC layer. Rotating the sample will result in a change in the azimuthal angle Φ, and will cause optical transmission to vary between minimum and maximum values alternatively. In the case of PNSFLCs, this principle has been applied in the examination of the molecular alignment of the host liquid crystal assuming that the presence of a low concentration of polymer network will not introduce an observable effect on the uniaxial structure of the host liquid crystal. In this investigation, a Nikon OPTIPHOT-POL polarizing microscope was used for the examination of the PNSFLC samples. The microscope was equipped with a rotating stage whose angular position could be set with an accuracy of ±0.1° using a vernier scale. In order to measure the transmittance of PNSFLC samples, one of the eye pieces of the microscope was fitted with a photodiode. A He-Ne laser was used as light source; this produced a light beam with a wavelength of 633 nm. The transmittance of the PNSFLC sample to the laser beam was detected by the photodiode. The output signal of the photodiode detector was monitored by a Hewlett-Packard 54602B oscilloscope.

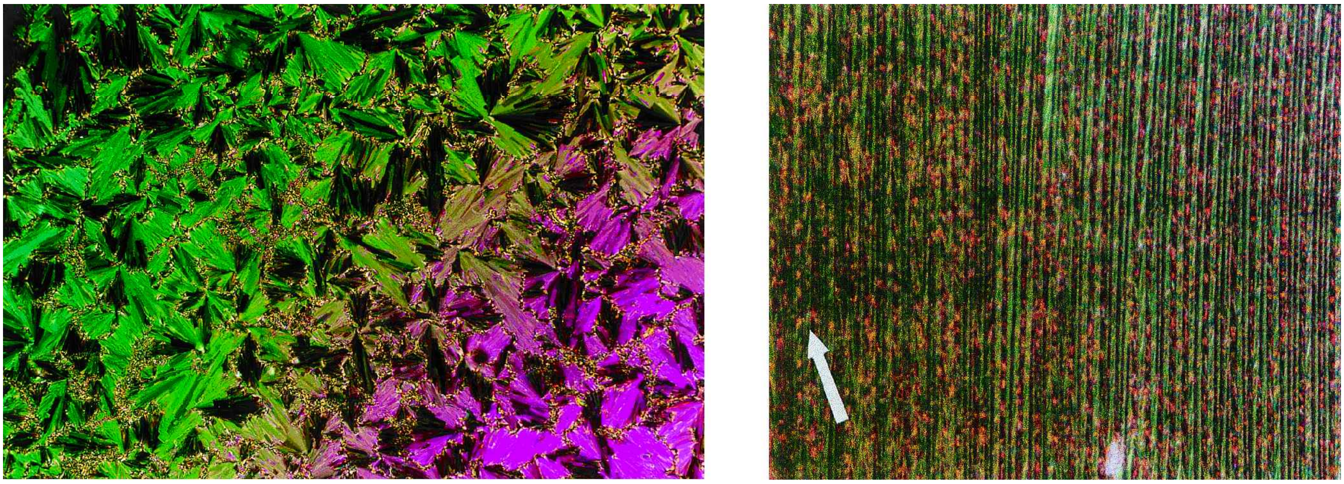
3. Results and discussion

3.1. *Preliminary observation*

A mixture was made by adding 3 wt % NOA65 to SCE3. The phase transition temperatures of the mixture were measured before UV curing the mixture, and the phase sequence of the mixture was found to be (°C):

$$\mathrm{SmC}^*\ 68.8\ \mathrm{SmA}\ 101.8\ \mathrm{N}^*\ 133.4\ \mathrm{I}.$$

In the presence of 3 wt % monomer, the phase transition temperatures for $\mathrm{SmC}^* \to \mathrm{SmA}$, $\mathrm{SmA} \to \mathrm{N}^*$ and $\mathrm{N}^* \to \mathrm{I}$ therefore dropped by 5.2, 3.2 and 2.6°C, respectively. A sample was made by introducing the mixture into an



(*a*) (*b*)

Figure 1. Photomicrographs showing PNSFLC layers made using SCE3/NOA65 composite with 3 wt % NOA65. The samples were cooled at a rate of 1°C min^{-1}, and the UV curing of the samples was carried out during the cooling process. (*a*) UV curing of the sample started from 65°C, and no magnetic field was applied during the UV curing. (*b*) The sample was cooled in the presence of a 4 kG magnetic field. The white arrow in the picture represents the direction of the magnetic field **H**. The UV curing of the sample started from 60°C. The photomicrographs were taken when the samples were at room temperature.

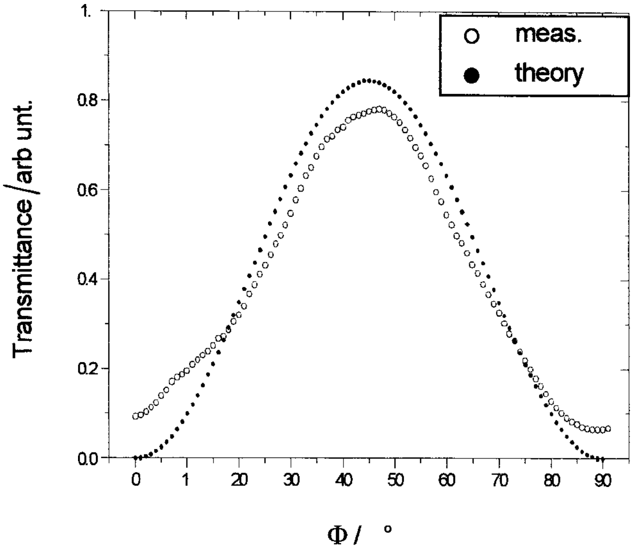


Figure 2. A comparison of the optical transmission of an aligned PNSFLC film with that of an ideally aligned SSFLC cell. Open circles represent the variation of optical transmission of the aligned PNSFLC film made using SCE plus 3 wt % NOA65 mixture as a function of the azimuthal angle. The thickness of the layer was 20 μm. The incident light was a He-Ne laser beam with a wavelength of 632.8 nm. The filled circles correspond to the theoretical transmittance of a 20 μm thick homogeneously aligned SCE3 layer. Data were obtained using formula (1). The birefringence of the liquid crystal was 0.17 (from Merck Ltd product data sheet).

alignment layer-free cell at a temperature of 150°C, and this was then cooled at a rate of 1°C min^{-1}. The sample was UV cured when it had cooled to 65°C, when the host liquid crystal was in the SmC* phase. The sample showed a focal-conic fun texture in the polarizing microscope, see figure 1 (*a*). This indicates that a homogeneous alignment of the PNSFLC has *not* been achieved in the sample cured without an external field. We then moved on to make another sample with the same composite material. In this case, the cell that was used had again no alignment layer. The mixture was introduced into the cell by capillary action at a temperature of 150°C. A magnetic field of 4 kG was applied, and the sample was cooled at a rate of 1°C min^{-1}. When it had cooled to 60°C, the sample was exposed to the UV light. The UV curing of the sample lasted for 60 min in the presence of the magnetic field while the sample was further cooled to ambient temperature. After UV curing, the sample was examined using the polarizing microscope where the PNSFLC layer showed a striped texture as shown in figure 1 (*b*). It was suggested that the striped texture of the SmC* phase could result from a special form of chevron structure in which chevons ran horizontally to the plane of the cell, and the defect lines ran along the smectic layer normal [13]. When the sample was rotated on the microscope stage, its appearance alternated between dark and bright. The transmittance of the sample was measured using the photodiode, and the data are plotted in figure 2. The transmittance of the sample became a maximum when $\Phi = (m \pm 1/2)\pi/2$ and a minimum when $\Phi = m(\pi/2)$, where m is an integer. The observations are in agreement with formula (1). This suggests that the molecules of the FLC in the cell are homogeneously aligned, i.e. the molecules of the FLC have a unique orientation. The transmittance of the

PNSFLC layer as a function of azimuthal angle was similar to that of a layer of homogeneously aligned FLC with the same thickness, except for a slight difference in intensity (cf. figure 2). The sample can modulate linearly polarized light in much the same way as a uniaxial birefringent liquid crystal layer.

The orientation of the liquid crystal molecules was not parallel to the direction of the magnetic field, but made an angle with it. The deviation angle between the average orientation of the liquid crystal molecules and the field direction can be measured by optical microscopy. In the sample discussed, the deviation angle was 18.7°.

3.2. *Effect of the concentration of NOA65 on the alignment of PNSFLCs*

The amount of polymer in the liquid crystal will determine the overall morphology of the composite. When the concentration of NOA65 was high, as expected a polymer dispersed ferroelectric liquid crystal (PDFLC) with liquid crystal microdroplets dispersed in the polymer matrix was obtained, after the polymerization of the polymer precursor. In the present studies, we have been unable to align a PDFLC using a magnetic field. Figure 3 shows a photomicrograph of a sample made using a composite with 10 wt % NOA65 in SCE3. The sample was UV cured in the 4 kG magnetic field when the sample temperature was 60°C. In the sample, the liquid crystal separated from the polymer binder and formed droplets. Since the amount of polymer was small, large liquid crystal domains were formed in the sample. When the sample was observed in the polarizing microscope, the liquid crystal in the droplets showed a conic fan texture.

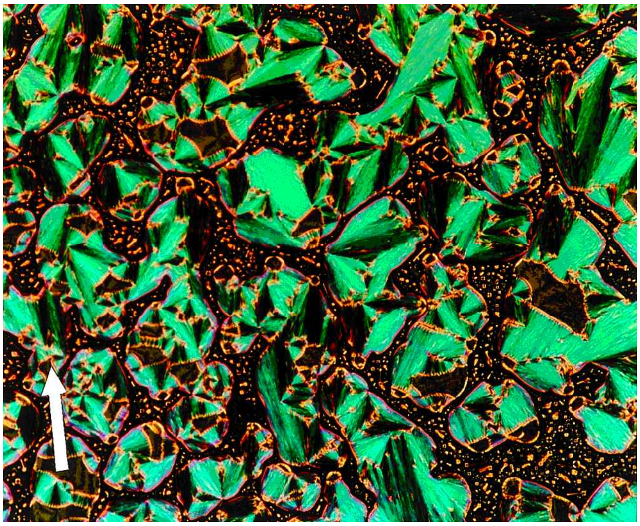


Figure 3. A photomicrograph showing a composite layer made using SCE3 plus 10 wt % NOA65 composite. The sample, with a thickness of 20 μm, was cooled to room temperature at a rate of 1°C $\min^{-1}$ in a 4 kG magnetic field. The white arrow represents the magnetic field direction. The UV curing of the sample was started from 60°C. The photomicrograph was taken when the sample was at room temperature.

Reducing the amount of NOA65 results in an increase in the size of the liquid crystal droplets. As the concentration of NOA65 decreases, it will become more and more difficult for the polymer to separate and bind the liquid crystal. When the concentration of NOA65 is sufficiently low, the polymer will no longer be able to separate and encapsulate the liquid crystal, but instead forms a web in the liquid crystal continuum. Figure 4 (*a*) shows a photomicrograph of a sample made using a composite containing 6 wt % NOA65. The sample was UV cured in the 4 kG magnetic field when the host liquid crystal was in the SmC* phase. In this sample, the continuum of NOA65 binder with microdroplets of liquid crystal has disappeared and is replaced by a polymer web immersed in the liquid crystal. The sample showed a focal-conic texture with a certain degree of orientation. This is certainly due to the effect of the polymer network. When the concentration of NOA65 was reduced to 4 wt %, the focal-conic fun texture in the PNSFLC layer became more oriented, cf. figure 4 (*b*). However, a homogeneously aligned PNSFLC layer was not obtained.

When the amount of the polymer precursor in the composite was further reduced, a PNSFLC layer with homogeneous alignment could be produced using the magnetic field. Figure 4 (*c*) shows a PNSFLC made using a composite consisting of 3 wt % NOA65. The sample was UV cured in the 4 kG magnetic field; UV curing of the sample was started at 60°C. The liquid crystal in the sample was homogeneously aligned.

A full explanation for the physical principle behind the experimental results has yet to be fomulated. The morphology of the polymer network is a crucial factor for the molecular alignment of the host liquid crystal. It has been shown that the polymer network formed in a liquid crystal environment contains nodular beads. If the size of the bead is small, the polymer network will have a fine fibrous structure. Such a polymer network favours alignment of the liquid crystal. Increasing the concentration of the monomer caused an increase in the size of the nodular beads [6], disturbing the molecular alignment of the host liquid crystal. In the present studies, the morphology of the polymer network has not been investigated. We believe that a high concentration, in this case > 4 wt %, of monomer in the composite will cause some significant change in morphology of the polymer network, and consequently violate the alignment of host liquid crystal.

3.3. *Effect of curing temperatures*

The polymerization of the polymer precursor was carried out by UV curing the sample during the cooling process. The UV curing of the sample can be started from different temperatures. When the temperature of the sample varies over a large range, the phase, i.e. the symmetry, of the host liquid crystal will be changed. We believed that the symmetry of the host liquid crystal in which the polymerization of the polymer precursor was carried out would certainly affect the morphology of the resultant polymer network. As the morphology of the polymer network changes, the orientation of the molecules and the geometrical structure of the molecules in the smectic layer of the host liquid crystal also change, and this will cause a corresponding change in texture of the liquid crystal; this can be observed by microscopy. In order to observe these variations, samples were prepared by starting the UV curing of the samples when their temperature was 60, 90, 110 and 140°C. Corresponding to these temperatures the host liquid crystal was in the SmC*, SmA, N*, and isotropic phase, respectively. The photomicrographs in figure 5 show PNSFLC layers which were produced by starting the UV curing from the different phases of the host liquid crystal. All the photomicrographs were taken when the samples were at room temperature, i.e. the host liquid crystal was in the SmC* phase. Starting UV curing of a sample at a temperature of 60°C, when the liquid crystal was in the SmC* phase, produced a PNSFLC layer with a striped texture, see figure 5 (*a*). As discussed before, the liquid crystal molecules in the sample are arranged in a chevron structure with chevrons running parallel to the cell plate, i.e. the horizontal chevron structure. The PNSFLC obtained with UV curing from a temperature of 80°C, when the host liquid crystal was in the SmA phase, also showed a striped texture. However, the width of the stripes was expanded, see figure 5 (*b*). Figure 5 (*c*) shows a PNSFLC layer produced by starting the UV curing of the monomer when the host liquid crystal was in the N* phase. In this PNSFLC layer, the striped texture disappears. The appearance of the zigzag defect lines suggests that the liquid crystal molecules in this layer are arranged in the chevron structure, with chevrons

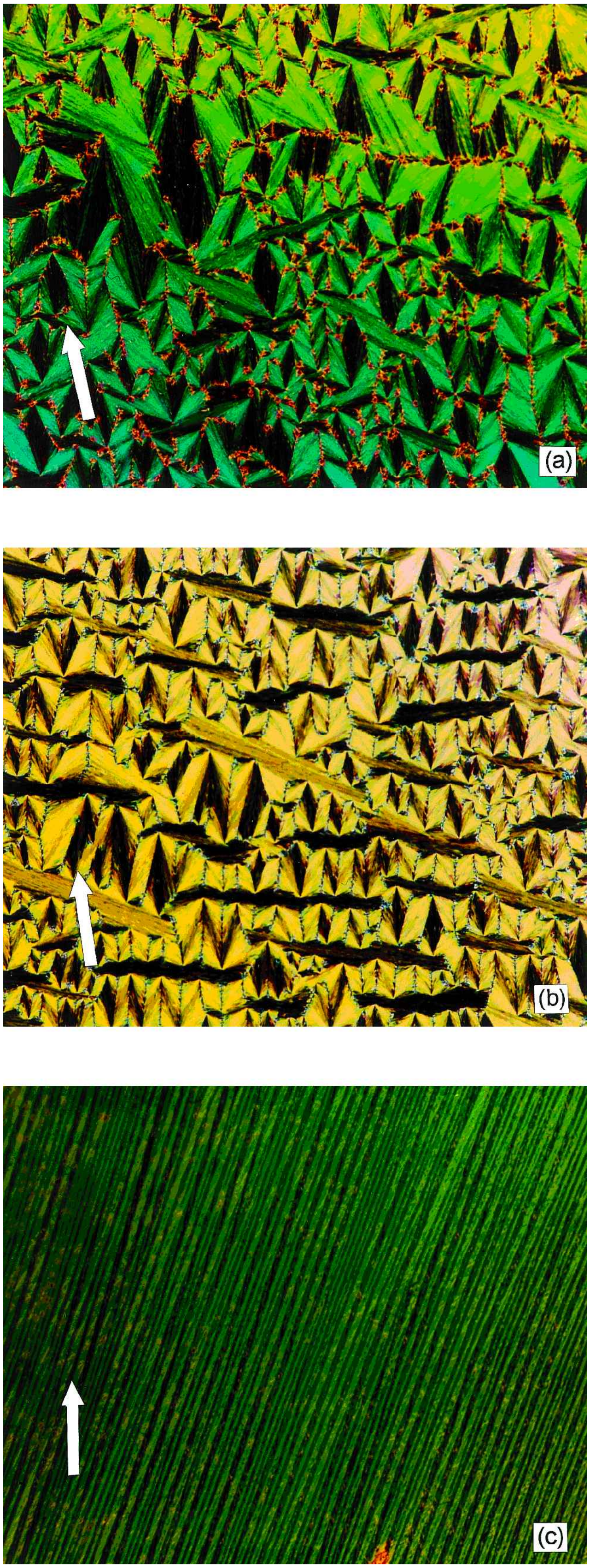


Figure 4. Photomicrographs showing PNSFLC layers in 20 μm thick cells at room temperature. Samples were cooled at a rate of 1°C min^{-1} in a 4 kG magnetic field. The UV curing of the samples was started at 60°C. The samples were made using SCE3 plus NOA65 composites. The concentrations of NOA65 in these samples are: (*a*) 6 wt %, (*b*) 4 wt % and (*c*) 3 wt %. The white arrow indicates the direction of the magnetic field. The photomicrographs were taken when the samples were at room temperature.

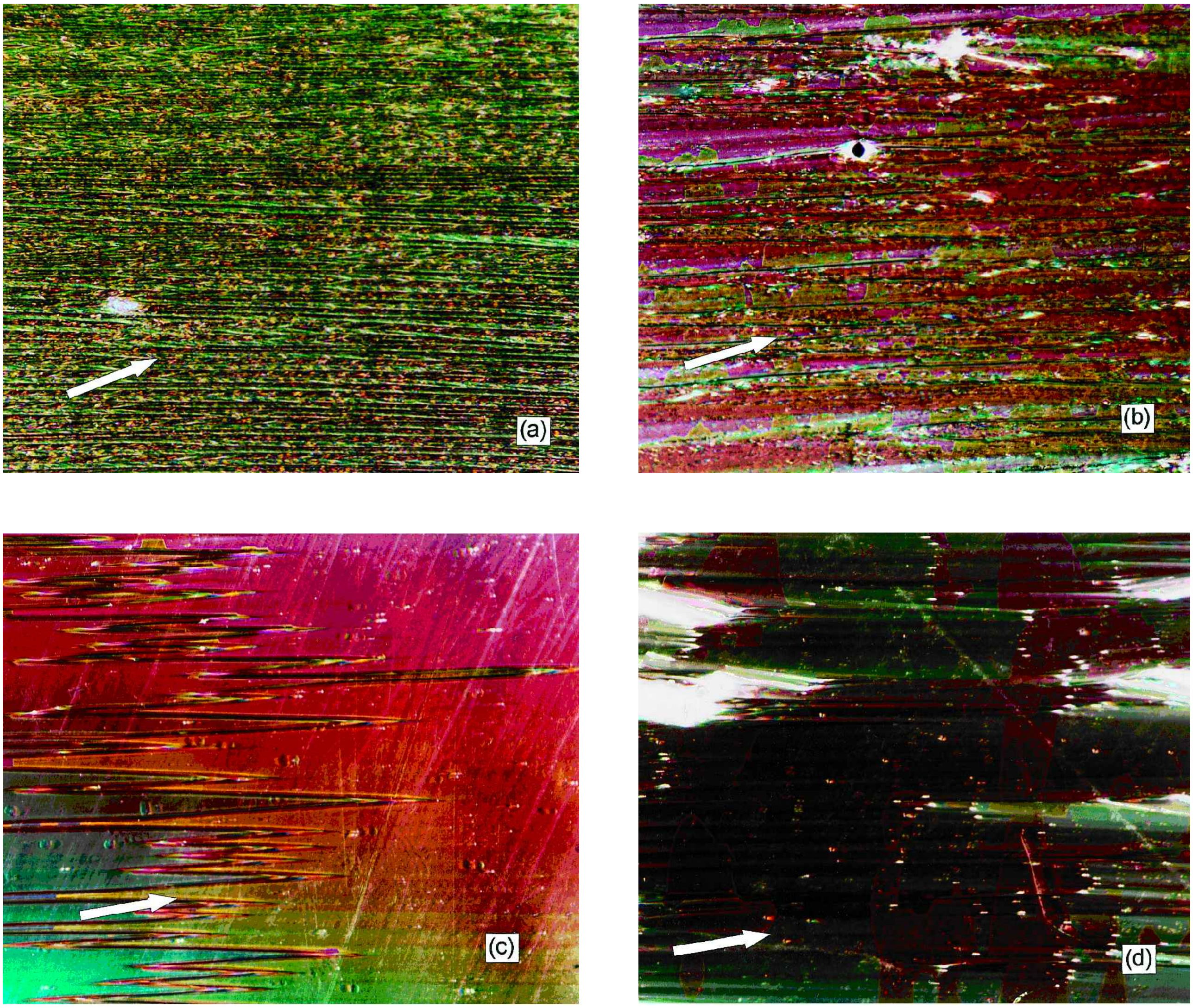


Figure 5. Photomicrographs showing PNSFLC layers produced by starting the UV curing of the samples when the host liquid crystal was in (*a*) SmC* phase (60°C), (*b*) SmA phase (90°C), (*c*) N* phase (110°C), and (*d*) isotropic phase (140°C). Samples were made using a SCE3 plus NOA65 composite consisting of 3 wt % NOA65, and cured in a 4 kG magnetic field. The white arrows in the pictures indicate the direction of the magnetic field. The photomicrographs were taken with the samples at room temperature.

running perpendicular to the cell plate, i.e. the vertical chevron structure.

It has been suggested that an aligned liquid crystal environment, in which the monomer is polymerized, is required for achieving an aligned PNSLC. In our investigation, it is interesting to note that in the presence of an external magnetic field the aligned liquid crystal environment may not be necessary. An aligned PNSFLC can be produced even if the monomer is polymerized in an isotropic environment. Figure 5 (*d*) shows a photomicrograph of a PNSFLC layer which was produced by starting the UV curing of the monomer when the host liquid crystal was in the isotropic phase. The sample is well aligned.

In the aligned PNSFLC films, the liquid crystal molecules, as mentioned in a previous section, do not orient in the direction parallel to that of the magnetic field. The orientation of the liquid crystal deviates from the direction of the magnetic field. It was found that starting the curing of the sample from different temperatures caused the deviation angle to vary. The deviation angle of orientation that the liquid crystal made with the direction of the magnetic field for PNSFLC samples was measured by optical microscopy. The measurements were carried out at room temperature, i.e. the host liquid crystal was in the SmC* phase. The results are given in the table. The deviation angles tend to increase as the starting temperature for the polymerization of the

Table. The features of PNSFLC films produced by starting the UV curing of the sample from different temperatures.

Starting temperature/°C	Layer thickness/μm	Texture	Deviation angle/°
60	20	Stripe	18.7
90	20	Stripe	15.5
110	20	Fine texture with zigzag lines	11.1
140	20	Fine texture with zigzag lines	6.4

monomer decreases. A smaller deviation angle was observed when the UV curing of the sample was started from a temperature at which the host liquid crystal was in a less ordered (i.e. more symmetrical) state, e.g. in the nematic or the isotropic phases.

The experimental results suggests that when the curing of the sample started from a temperature at which the host liquid crystal was in the SmC* or the SmA phase, the liquid crystal molecules in the PNSFLC layer could be arranged in a horizontal chevron structure, and the deviation between the orientation of the liquid crystal molecules and the direction of the magnetic field was large (up to 18.7°). If the polymerization of the monomer was started when the host liquid crystal was in the nematic state, the liquid crystal molecules could be arranged in the vertical chevron structure, and a smaller deviation angle between the molecular director of the liquid crystal and the direction of the magnetic field was observed. The chevron structure of SmC* phase results from the balance between the surface anchoring and the shrinkage of the smectic layers.

In our case, the presence of the polymer network will certainly influence the configuration of the host liquid crystal. In samples which were produced from the polymerization of the monomer in the isotropic phase or the nematic phase, the polymer network was formed before the formation of the smectic layers of the liquid crystal. Both the polymer network and the magnetic field will impose actions on the liquid crystal. As a result, the smectic layer of the host liquid crystal shrank to form a vertical chevron structure. In the nematic phase, the liquid crystal molecules may be aligned by the magnetic field to orient in the direction of the magnetic field, and anchored to the polymer network. The alignment of the liquid crystal was then sustained by the polymer network. This results in a small deviation of the liquid crystal director from the magnetic field direction, i.e. the initial orientation direction of the liquid crystal in the nematic phase.

In those samples which were produced by starting the UV curing of the sample after the host liquid crystal had changed into the layered mesophase(s), the crosslinking of the monomer took place after smectic layer formation. When the smectic layers were shrinking, there was no polymer network. The major effective action on the liquid crystal was from the magnetic field. The smectic layers shrank to form a horizontal chevron structure in the presence of the magnetic field. After polymerization of the monomer, a polymer network formed, and it, in turn, froze the alignment of the host liquid crystal.

4. Conclusions

It has been shown that PNSFLC layers with a homogeneous alignment can be produced using a magnetic field. In this technique, the use of surface alignment layers is not necessary, and the surface treatment for the substrates can be simplified. The use of a magnetic field to align ferroelectric liquid crystals was demonstrated two decades ago, and it was pointed out that to achieve a unique molecular orientation in a pure FLC, a strong field (> 1 T) was demanded [14]. It was found that, in the presence of a small amount of a suitable monomer, it was easier to align the FLC using a magnetic field. In the present studies, we have successfully produced homogeneously aligned PNSFLCs using a 4 kG magnetic field. In our experiments, an even smaller (~ 2 kG) magnetic field was tried, and a well aligned PNSFLC layer was produced [15].

The amount of the polymer precursor in the FLC is crucial for molecular alignment of the FLC/polymer composite, and is determined by the liquid crystal and the polymer precursor used. For the SCE3/NOA65 system, the concentration of the polymer precursor must not be higher than 4 wt % in order to achieve a homogeneously aligned PNSFLC layer using a 4 kG magnetic field.

In PNSFLC layers, which were produced by starting the polymerization of the monomer from different temperatures, at which the host liquid crystal was in different phases, the liquid crystal molecules may be arranged in different geometrical structures. In most PNSFLC samples which were produced in the present studies, the FLC molecules in the SmC* phase are packed in a chevron structure. However, the chevrons will tend to run parallel to the cell plate if the monomer is polymerized in the nematic environment, or to run perpendicular to the cell plate if the monomer is crosslinked in the smectic environment. It was however found that an aligned liquid crystal environment prior to the polymerization of the monomer is not necessarily a prerequisite condition for achieving an aligned PNSFLC layer. A homogeneously aligned PNSFLC layer can be produced by polymerizing the monomer in the isotropic phase. The mechanism behind this experimental result is still not understood.

It was also found that in a smectic polymer network system aligned by a magnetic field, the orientation of the liquid crystal tends to deviate from the magnetic field direction. The deviation angle between the liquid crystal director and the magnetic field direction was increased when UV curing of the sample was started from a temperature at which the host liquid crystal was in a more ordered phase.

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