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Direct decoration of disclinations by solidification-induced band texture and focal-conic texture for a low molar mass liquid crystal

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Two decoration methods, solidification-induced band texture decoration and focal-conic texture decoration, were established to map the director field of disclinations in a low molar mass methacrylate liquid crystal. It was found that when the specimen film is quenched from the nematic melt to room temperature, solidification-induced band texture is observed arrayed along with the schlieren texture, and the orientation of the director field can be displayed. Moreover, when the specimen is cooled from the nematic melt to 63°C and annealed, the focal-conic texture of the smectic A phase is found to grow around the corresponding disclination core with good orientation to reveal the director field. By the two decoration techniques, the director fields of disclinations with strength $s = 1/2$ and $s = \pm 1$ were revealed. Two types of inversion wall, loop-like wall and splay-type wall, were found by both solidification-induced band texture decoration and focal-conic texture decoration.

1. Introduction

The disclination is a common phenomenon in liquid crystals. It is the discontinuity of the director field in space [1]. In 1958 Frank schematized the mapping of the corresponding director arrangement of molecules according to the disclination theory of low molar mass liquid crystals (LMMLC) [2]. Thereafter various methods have been developed to detect experimentally the arrangement of the molecular director in the vicinity of disclinations. So far, four kinds of method, lamellar decoration [3, 4], band texture decoration [5], surface microcrack decoration [6–8] and focal-conic texture decoration [9], have been established to observe the discontinuity in the director field of liquid crystalline polymers (LCPs). However, for low molar mass liquid crystals there are only two examples of decoration for disclinations. One is the gas bubble decoration method reported by Cladis *et al.* [10] to display the director field of 4-methoxybenzylidene-4′-*n*-butylaniline (MBBA). In this method a solution of MBBA in diethylene glycol is spread on a glass slide to form a thin film of several μm thickness during the process of solvent evaporation; at the same time a lot of gas bubbles of 3–4 μm diameter are formed and apparently arranged equidistantly into long chains along the direction of the director of the MBBA molecules. The other decoration technique proposed by Rault [11] is used to reveal the director fields of MBBA samples by microcrystallite decoration; in this method poly(benzyl-L-glutamate) (PBLG) microcrystallites are organized into chains of a few microns width along the local director of the MBBA molecules and so the director field of the MBBA is revealed.

Until now, however, no other decoration method has been reported for observing the director field of low molar mass liquid crystals directly. In the present work, the solidification-induced band texture and focal-conic texture are observed arrayed along with the schlieren texture in the glassy state of a low molar mass methacrylate liquid crystal. On this basis, direct observation of the director orientation image decorated by the solidification-induced band texture or focal-conic texture was realized and discussed.

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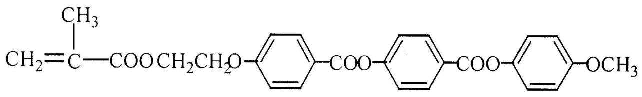
2. Experimental

The low molar mass methacrylate liquid crystal was synthesized in our laboratory and was denoted MA-2. The chemical structure is shown below.



The molecular mass of MA-2 is 476. The sample showed a nematic-to-smectic A transition at 87°C ($T_{N\text{-}Sm}$) and a smectic to crystal transition at 61°C ($T_{Sm\text{-}Cr}$), determined by differential scanning calorimetry (DSC-7, Perkin-Elmer).

First, the powder sample was pressed into a thin film between two glass slides at 110°C. Specimens for observation of the solidification-induced band texture were prepared by holding the thin film at 110°C for 2 min and then quenching to room temperature as quickly as possible. Specimens for observation of the focal-conic texture were obtained by cooling the thin films from 110 to 63°C at a rate of 10°C min^{-1}. The morphological observation of the solidification-induced band texture and focal-conic texture was carried out by polarizing optical microscopy (POM) (Olympus BH-2, Japan, equipped with a Mettler FP-52 hot stage and an automatic camera).

The smectic phase was characterized as SmA from the microscopic textures and from the WAXD layer spacing of 27.25 Å corresponding with the molecular length in the all-*trans*-conformation of 27.55 Å.

The specimens for infrared dichroism measurement were prepared by pressing the powder sample into a thin film between a NaCl plate and a PTFE film at 110°C and then removing the PTFE film. The specimen on the NaCl plate was kept at 110°C for 2 min and subsequently cooled to 63°C to form the focal-conic texture. Eventually the specimen was quenched to room temperature. Infrared dichroism measurements of the specimen were performed using a Fourier Transform IR spectrometer (Bruker IFS-113V) with a Perkin-Elmer gold wire grid polarizer at a resolution of 2 cm^{-1}.

3. Results and discussion

3.1. *Solidification-induced band texture decoration*

The solidification-induced band texture is usually formed by quenching main chain liquid crystalline polymers from the nematic melt to room temperature [12, 13]. It is commonly thought that such a band texture cannot be formed in low molar mass liquid crystalline materials. However, it has been found that the solidification-induced band texture can be observed in our low molar mass specimen MA-2 when quenching is carried out from 110°C to room temperature. Figure 1 shows the POM micrograph of the solidification-induced band texture of MA-2 and it is found that the band texture is arrayed along with the schlieren texture formed by the nematic melt. The width of the solidification-induced band is about 1 μm. It can be seen that there are three disclinations with strength $s = |1|$ in figure 1. The type of the disclination can be determined by the pattern of the band texture around a disclination if the band direction is closely related to the molecular director.

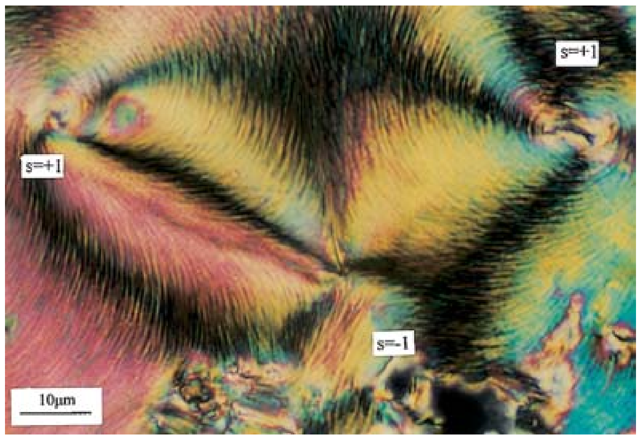
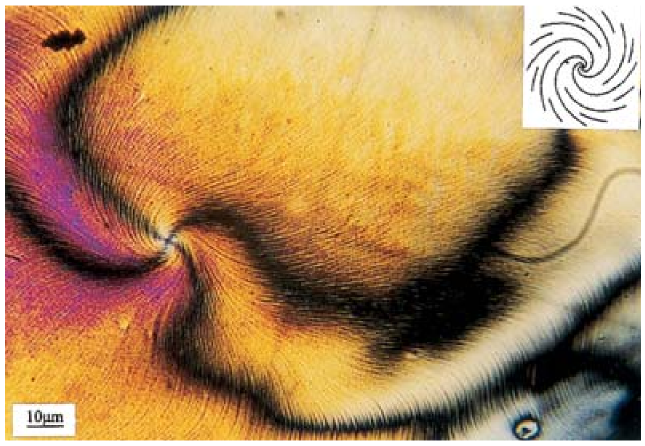


Figure 1. Disclinations with $s = |1|$ decorated by the solidification-induced band texture.

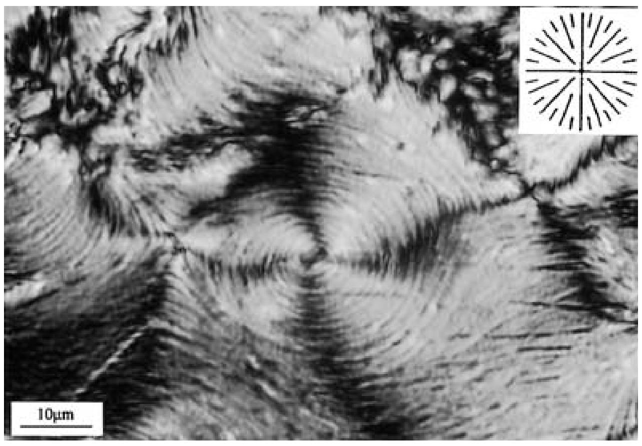
In order to reveal the director field around the disclinations and to determine their type, the relationship between the direction of the band orientation and the director orientation must be ascertained. It has been proved that the band direction in the solidification-induced band texture is perpendicular to the direction of the molecular director for main chain liquid crystalline polymers. Since the low molar mass liquid crystal MA-2 can be regarded as a short main chain liquid crystal, it is believed that the direction of the director field is perpendicular to the band direction. In this way the types of the disclinations can be determined. The disclination strengths in figure 1 are marked near the corresponding disclinations.

Figure 2 shows the POM micrographs of different types of disclinations decorated by the solidification-induced band texture in MA-2. The disclinations with $s = \pm 1$ are found to be the main defects, figures 2 (*a–d*), with occasionally the disclination with $s = +1/2$, figure 2 (*e*). However, no disclination with $s = -1/2$ has been observed up to now in the MA-2 sample. In the $s = +1$ case, there are three types of director orientation corresponding to different angles between the orientation axis of the molecular director and the main optical axis (angle *c*). The director fields of the different disclinations are mapped at the top right of the micrographs in figure 2. The three types of disclination for $s = +1$ can be distinguished

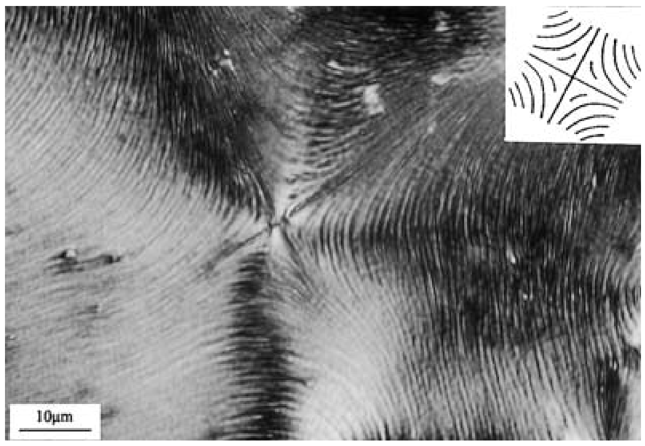
(*a*)



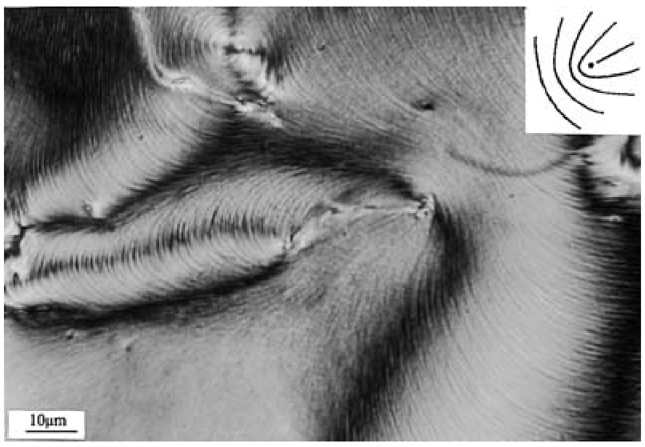
(*b*)



(*c*)



(*d*)

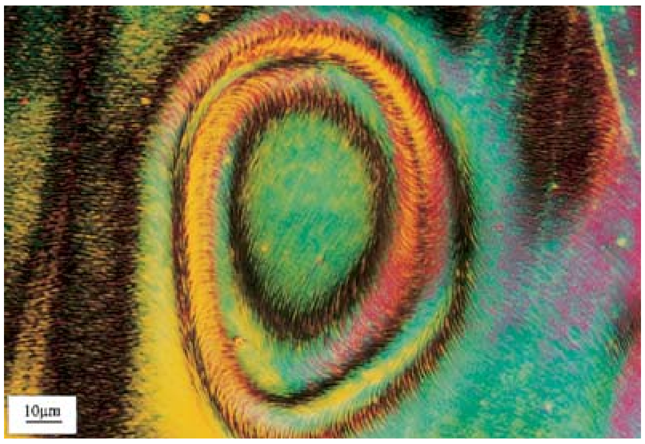


(*e*)

Figure 2. Disclinations with strength $s = +1$ and (*a*) $c = 0$, (*b*) $c = \pi/3$, (*c*) $c = \pi/2$, as well as (*d*) $s = -1$ and (*e*) $s = 1/2$ decorated by the solidification-induced band texture.

according to the angle $c = 0$, $c = \pi/3$ and $c = \pi/2$ shown in the sketch map of the director field. That is, figure 2 (*a*) corresponds to the angle $c = 0$, figure 2 (*b*) to the angle $c = \pi/3$, and figure 2 (*c*) to the angle $c = \pi/2$.

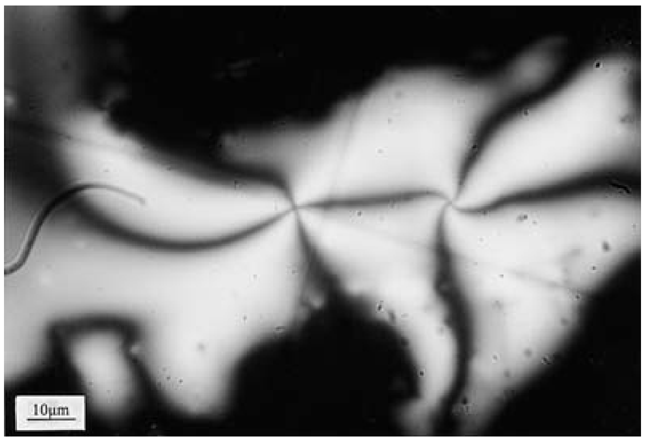
The inversion wall is one kind of defect found in the liquid crystalline state. There are two types of inversion wall observed in MA-2, viz. the loop-like wall, figure 3 (*a*), and the splay-type wall, figure 3 (*b*). Both types of wall



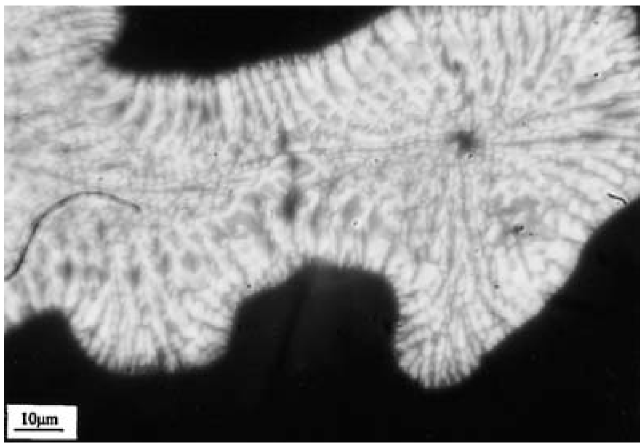
(*a*)

(*b*)

Figure 3. Two types of inversion wall decorated by the solidification-induced band texture: (*a*) loop-like wall, (*b*) splay-type wall.



(*a*)



(*b*)

Figure 4. Nematic schlieren texture at 110°C (*a*) and corresponding focal-conic texture formed at 63°C (*b*).

are decorated by solidification-induced band texture. It is suggested that the molecular director is turned through an angle π across the singularity line for both types of wall.

3.2. *Focal-conic texture decoration*

Usually the focal-conic texture can be used to decorate the disclinations formed in the nematic melt and therefore it is required that the liquid crystalline material shows the phase transition from nematic to smectic. Such a decoration technique has been applied to a side group liquid crystalline polymer by Hu *et al.* [9].

For MA-2 there are two mesophases, nematic and smectic A, and so it is possible to decorate the disclinations formed in the nematic phase by the focal-conic texture. Figure 4 shows the POM micrographs of the liquid crystalline states of MA-2 at different temperatures. It can be seen from figure 4 (*a*) that the typical schlieren texture of the nematic phase is observed by POM in the MA-2 specimen held at 110°C for 2 min. However, when the temperature is decreased to 63°C, taking the existing defects as a template, the typical focal-conic texture of the smectic A mesophase grows around the nematic schlieren texture core, see figure 4 (*b*). The pattern of the director field of the liquid crystal of MA-2 can therefore be displayed by the focal-conic texture growing around the core of a disclination.

The director fields of different types of disclinations decorated by the focal-conic texture are shown in figure 5. The disclination with $s = 1/2$ is observed by the focal-conic texture decoration, figure 5 (*a*). However, a disclination with $s = -1/2$ cannot be found in MA-2. The disclinations with $s = \pm 1$ are illustrated in figures 5 (*b–e*). It is clear that there is alignment of the disclination with $s = -1$ in figure 5 (*b*). Figures 5 (*c–e*) are three types of disclination with $s = +1$, corresponding to different angles *c*. However, the precise director orientation of the different disclinations cannot be ascertained by POM observation only.

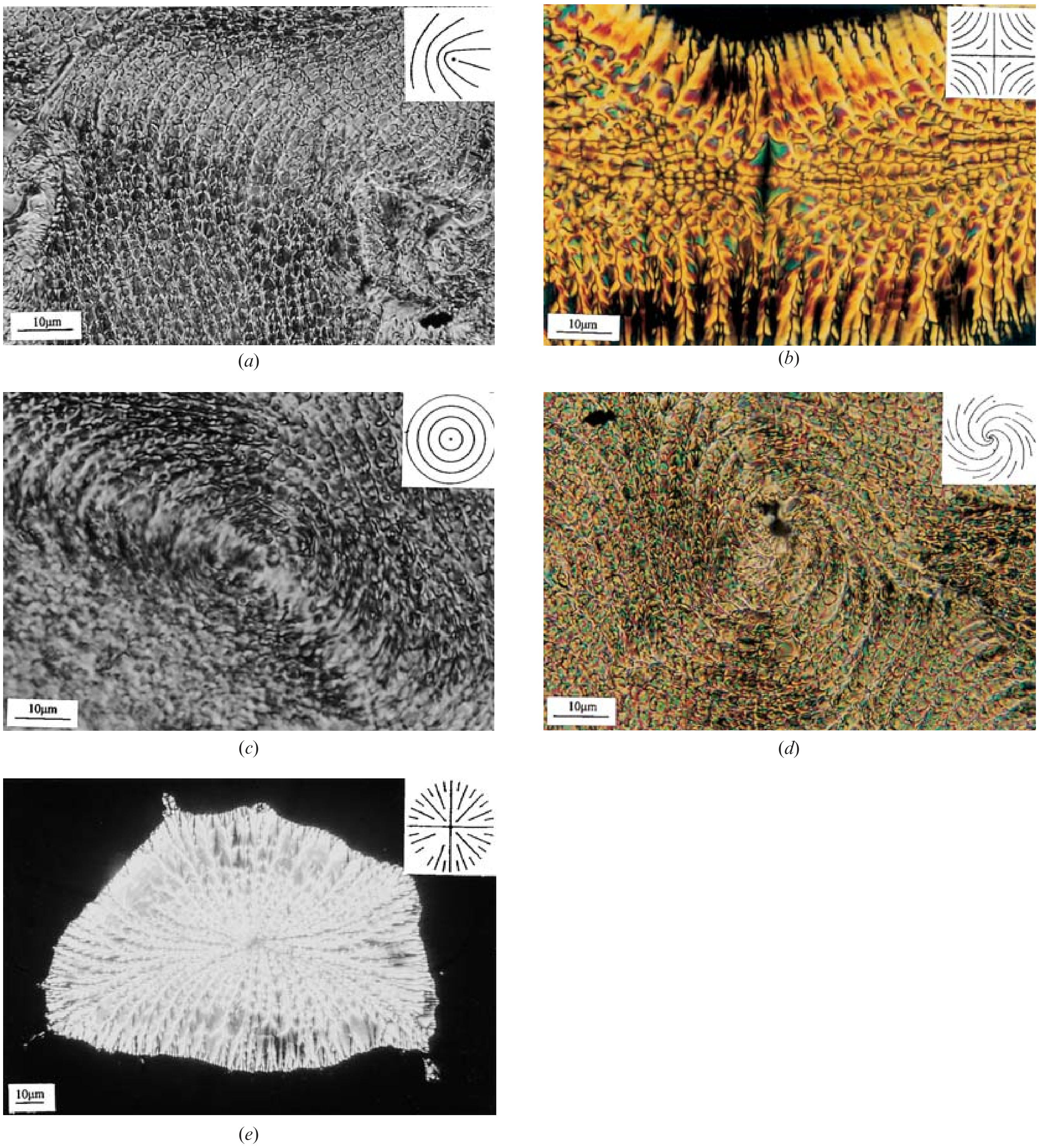
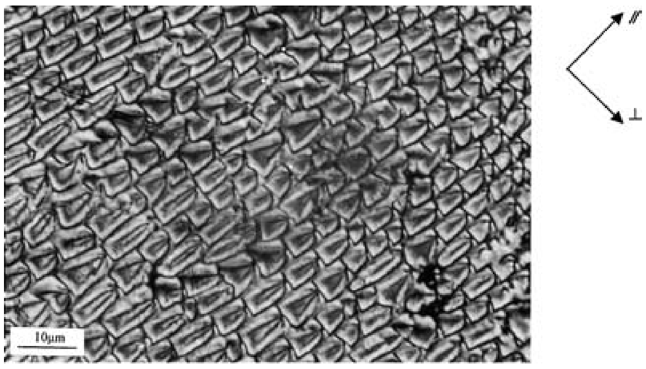
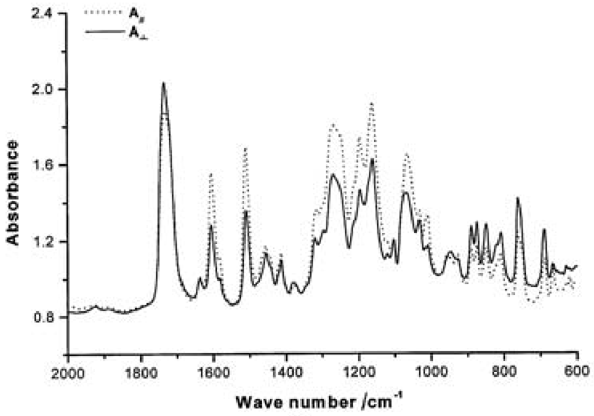


Figure 5. Disclinations with the strength (*a*) $s=1/2$, (*b*) $s=-1$, (*c*) $s=+1$ and $c=0$, (*d*) $s=+1$ and $c=\pi/3$, (*e*) $s=+1$ and $c=\pi/2$ decorated by the focal-conic texture.

In order to map the director field and determine the type of the disclination, unambiguous information about the relationship between the direction of the focal-conic orientation and the direction of the molecular director must be determined. Polarizing FTIR spectroscopy is thought to be an effective technique [14]. The IR



(a)



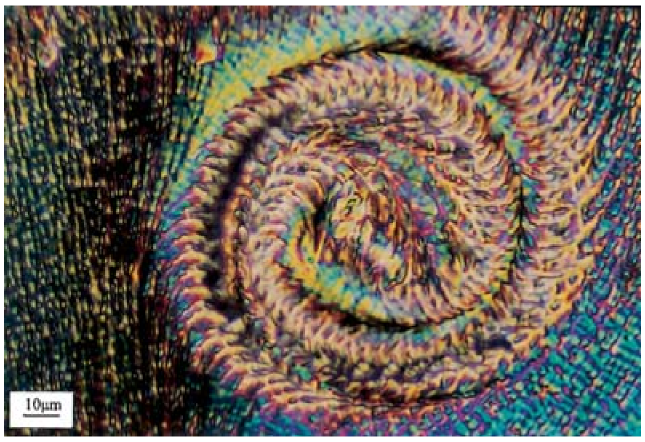
(b)

Figure 6. (*a*) Oriented region of the focal-conic texture; (*b*) polarized IR spectra of the oriented region shown in (*a*).

dichroism spectrum of the well ordered region shown in figure 6 (*a*) is illustrated in figure 6 (*b*), where $A_{\parallel}$ and $A_{\perp}$ are the absorbances for radiation polarized parallel and perpendicular to the direction of focal-conic texture orientation, respectively. The main characteristic absorption bands and the values of the dichroic ratios are given in the table. The C−O−C stretching bands at 1064, 1161, 1196 and 1267 cm^{-1}, as well as the phenylene ring stretching bands at 1510 and 1604 cm^{-1}, exhibit distinct parallel dichroism, $A_{\parallel}/A_{\perp} > 1$. However, the out-of-plane vibrations of the phenylene ring C−H bending and phenylene ring bending at 762 and 688 cm^{-1}, respectively, exhibit perpendicular dichroism, $A_{\parallel}/A_{\perp} < 1$. Because the C−O−C and the phenylene ring stretching vibrations have transition moments parallel to the direction of the axes of the mesogen, the mesogen is arrayed parallel to the orientation direction of the focal-conic texture; that is, the direction of the MA-2 molecule is parallel to the orientation direction of the focal-conic texture. According to the results of the IR dichroism measurements, therefore, the director field of the disclination can be revealed by the morphological patterns of the focal-conic texture shown in figure 5; the director fields are mapped in the top right of the corresponding POM micrographs on the figure. Different types of disclinations with $s = +1$ in figures 5 (*c*–*e*) corresponding to the angles $c = 0$, $c = \pi/3$ and $c = \pi/2$, respectively, can also be distinguished.

Table. Some IR absorption band assignments and values of their dichoric ratios for MA-2.

Frequency/cm^{-1}	Tentative assignment	$R = A_{\parallel}/A_{\perp}$
688	Out-of-plane phenylene ring bending	<1
762	Out-of-plane phenylene ring C−H bending	<1
1064	C−O−C stretching	>1
1161	C−O−C stretching	>1
1196	C−O−C stretching	>1
1267	C−O−C stretching	>1
1510	Phenylene ring stretching	>1
1604	Phenylene ring stretching	>1



(*a*)

(*b*)

Figure 7. Two types of inversion wall decorated by the focal-conic texture: (*a*) loop-like wall, (*b*) splay-type wall.

As shown in figures 7(*a*) and 7(*b*), the two types of inversion wall (i.e. the loop-like wall and the splay-type wall) are also revealed by the focal-conic texture decoration.

4. Conclusions

Solidification-induced band texture is observed by POM when the low molar mass liquid crystal MA-2 is quenched from the nematic phase to room temperature. Also, when the specimen is cooled from the nematic melt to 63°C, the focal-conic texture is formed. Both decoration techniques, the solidification-induced band texture and the focal-conic texture decoration, are applied to reveal the orientation images of the director fields for MA-2. The director fields of the disclinations with strengths $s = 1/2$, $+1$, and -1 are displayed by the two decoration methods. At the same time, two types of inversion wall, loop-like wall and splay-type wall, can also be observed by the solidification-induced band texture and the focal-conic texture decoration.

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