Synthesis and Mesomorphic Properties of a New Side-Chain, Chiral Smectic, Liquid-Crystalline Elastomer

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ABSTRACT: The synthesis of the new chiral monomer 4-(10-undecylen-1-yloxy)biphenyl-4′-[(*S*)-2-methyl-1-butoxy]benzoate (**M$_1$**), the nematic crosslinking agent biphenyl 4,4′-bis(10-undecylen-1-yloxybenzoate) (**M$_2$**), and the corresponding liquid-crystalline elastomer is described. The chemical structures of the chiral monomer and crosslinking agent have been characterized with Fourier transform infrared, elemental analyses, and proton and carbon-13 nuclear magnetic resonance spectra. The mesomorphic properties have been investigated with differential scanning calorimetry, polarizing optical microscopy, and X-ray diffraction. Monomer **M$_1$** shows different smectic phases (smectic A, chiral smectic C, and smectic B) and a cholesteric phase, and **M$_2$** exhibits a nematic phase. The liquid-crystalline elastomer shows smectic A and chiral smectic C phases. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 100: 4234–4239, 2006

Key words: chiral; elastomers; synthesis

INTRODUCTION

Since the synthesis of liquid-crystalline elastomers (LCEs) was reported in 1981,[1] research into LCEs as a new class of liquid-crystalline (LC) materials has expanded rapidly because they give rise to macroscopic features.[1–16] LCEs combine the basic features of polymer networks with the anisotropy of LC physical properties. Therefore, LCEs not only have entropic elasticity but also show reversible LC phase transitions during heating and cooling cycles. In particular, chiral LCEs have attracted considerable interest because of their remarkable electromechanical properties, such as piezoelectricity.[17–28] This has led to advanced applications in, for example, nonlinear optical devices, full-color thermal imaging, and electrooptic materials. Chiral LCEs have the potential to act as devices that transform a mechanical signal into an electric signal, and they are considered candidates for new piezoelectric materials. From a scientific point of view, cholesteric or ferroelectric [chiral smectic C* (S_C*)] LCEs are fascinating because they allow a study of the interplay of electric and mechanical forces in a rubbery material. This happens because the reorientation of the mesogenic groups in the electric field creates stress in the network of the polymer chains.

The aims of our research were (1) to prepare new chiral LCEs containing different crosslinking agents, (2) to study the structure–property relationships of side-chain chiral LCEs, and (3) to further explore their applications as novel piezoelectric devices. A previous study reported the synthesis and characterization of side-chain, cholesteric LCEs.[29–33] In this study, a new side-chain, chiral smectic LCE derived from a nematic crosslinking agent was prepared. The mesomorphic properties and phase behavior of the monomers and elastomer were characterized with polarizing optical microscopy (POM), differential scanning calorimetry (DSC), and X-ray diffraction (XRD).

EXPERIMENTAL

Materials

Polymethylhydrosiloxane (PMHS; number-average molecular weight = 700–800) was purchased from Jilin Chemical Industry Co. (Jilin, China). Undecylenic acid was purchased from Beijing Jinlong Chemical Reagent Co., Ltd. (Beijing, China). 4,4′-Dihydroxybiphenyl (Aldrich) was used as received. The H_2PtCl_6 catalyst was obtained from Shenyang Chemical Reagent Co. (Shenyang, China). All solvents and reagents were purified by standard methods.

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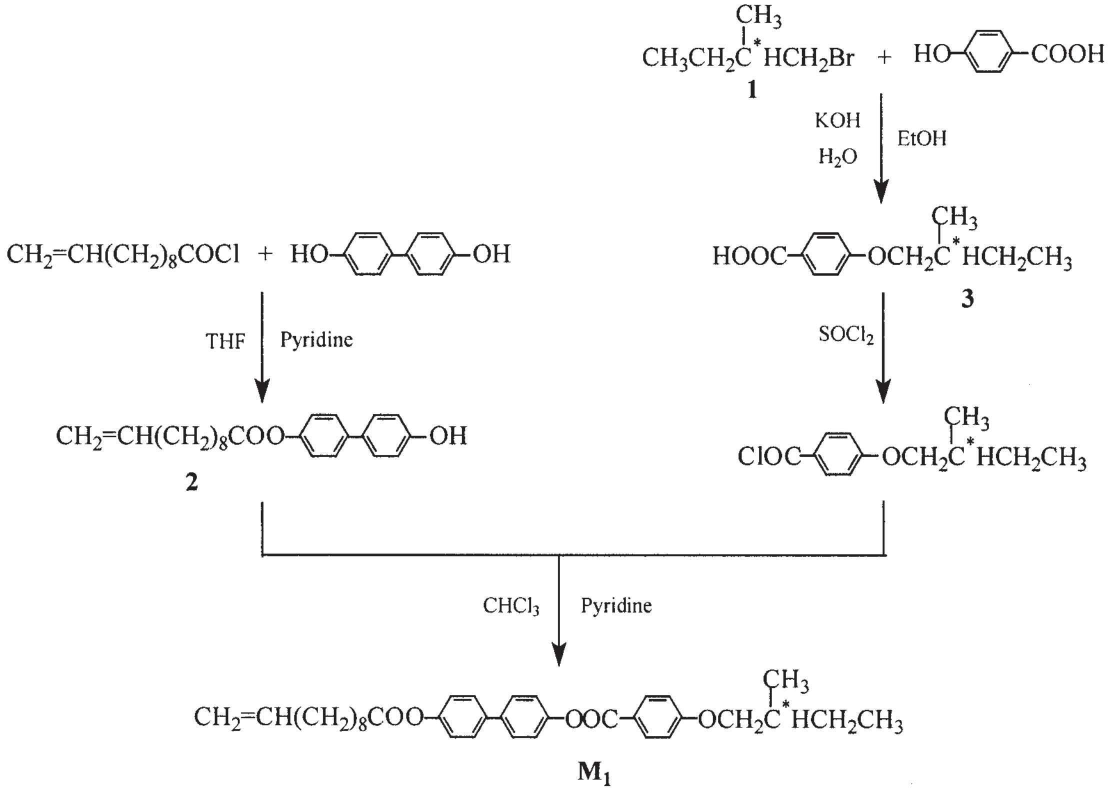


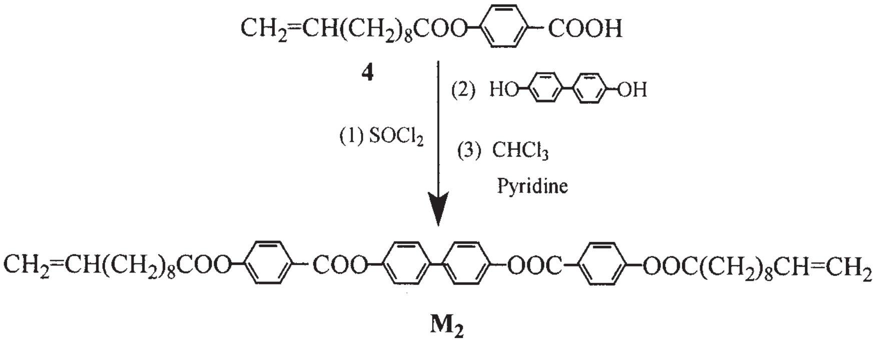
Scheme 1 Synthetic route of the LC monomer.

Measurements

Fourier transform infrared spectra were measured on a Spectrum One (B) spectrometer (PerkinElmer, Foster City, CA). The elemental analyses were carried out with a Elementar Vario EL III (Elementar, Hanau, Germany). Proton nuclear magnetic resonance (^{1}H-NMR) spectra (300 MHz) and carbon-13 nuclear magnetic resonance (^{13}C-NMR) spectra (75.4 MHz) were obtained with a Gemini 300 spectrometer (Varian Associates, Palo Alto, CA). Optical rotations were obtained on a PerkinElmer 341 polarimeter. Phase-transition temperatures and thermodynamic parameters were determined with a DSC 204 (Netzsch, Hanau, Germany) equipped with a liquid-nitrogen cooling system. The heating and cooling rates were 10°C/min. A DMRX POM instrument (Leica, Germany) equipped with a THMSE-600 hot stage (Linkam, England) was used to observe phase-transition temperatures and analyze LC properties through the observation of optical textures. XRD measurements were performed with nickel-filtered Cu Kα (λ = 1.542 Å) radiation with a DMAX-3A powder diffractometer (Rigaku, Tokyo, Japan).

Synthesis of the monomers

The synthesis of the olefinic monomers is shown in Schemes 1 and 2. The compounds (*S*)-(+)-2-methyl-1-



Scheme 2 Synthetic route of the crosslinking agent.

bromobutane (**1**) and 4-(10-undecylen-1-yloxy) benzoic acid (**4**) were prepared according to literature methods.[31,34]

4-hydroxybiphenyl-4′-(10-undecylen-1-ate) (**2**)

Undecylenyl chloride (20.4 g, 0.1 mol) was dissolved in 20 mL of tetrahydrofuran (THF) and then added dropwise to a solution of 4,4′-dihydroxybiphenyl (93.0 g, 0.5 mol) in 300 mL of THF and 8 mL of pyridine. After the solution was refluxed for 8 h, the reaction mixture was poured into a beaker filled with 500 mL of water; the crude product was obtained by filtration and recrystallized from acetone/ethanol (1:2). A white solid (**2**) was obtained.

Yield: 81%. mp: 124°C. IR (KBr): 3345 (—OH); 3085 (═C—H); 2973, 2852 (—$\mathrm{CH_2}$—); 1756 (C═O); 1641 (C═C); 1605, 1503 $\mathrm{cm^{-1}}$ (Ar—). ANAL. Calcd for $\mathrm{C_{23}H_{28}O_3}$: C, 78.37%; H, 8.01%. Found: C, 78.25%; H, 7.92%. ^{1}H-NMR [$\mathrm{CDCl_3}$, tetramethylsilane (TMS), δ, ppm]: 1.23–1.28 [s, 10H, —$(C\mathit{H}_2)_5$—]; 1.51–1.90 (m, 4H, —$C\mathit{H}_2\mathrm{CH_2COO}$— and $\mathrm{CH_2}$═$\mathrm{CH}C\mathit{H}_2$—); 2.21 (t, 2H, —$C\mathit{H}_2\mathrm{COO}$—); 4.98 (dd, 1H, $C\mathit{H}_2$═CH—); 5.04 (dd, 1H, $C\mathit{H}_2$═CH—); 5.75 (m, 1H, $\mathrm{CH_2}$═C*H*—); 6.77 (dd, 2H, Ar—*H*); 7.15 (dd, 2H, Ar—*H*); 7.30 (dd, 2H, Ar—*H*); 7.48 (dd, 2H, Ar—*H*); 5.2 (s, 1H, —O*H*).

4-[(*s*)-2-methyl-1-butoxy] benzoic acid (**3**)

4-Hydroxybenzoic acid (15.4 g, 0.12 mol) was dissolved in a mixture of 80 mL of ethanol, potassium hydroxide (16.8 g, 0.3 mol), and a small amount of potassium iodide in 32 mL of water. After being cooled to room temperature, compound **1** (15.1 g, 0.1 mol) was added dropwise to the solution. The reaction mixture was refluxed for 12 h and cooled to room temperature and then was diluted with water and neutralized with 20% chlorine hydride. The precipitated product was filtered and washed with hot water. The crude product was recrystallized from ethanol. A white crystal was obtained.

Yield: 83%. mp: 97°C. α_D^{20}: +0.26° (toluene). IR (KBr): 2924, 2853 (—$\mathrm{CH_3}$, —$\mathrm{CH_2}$—); 2642, 2530 (—COOH); 1671 (C═O); 1606, 1501 (Ar—); 1255 $\mathrm{cm^{-1}}$ (C—O—C). ANAL. Calcd for $\mathrm{C_{12}H_{16}O_3}$: C, 69.21%; H, 7.74%. Found: C, 69.32%; H, 7.92%. ^{1}H-NMR ($\mathrm{CDCl_3}$, TMS, δ, ppm): 0.92–1.10 (m, 6H, —$C\mathit{H}_3$); 1.31–2.13 (m, 3H, >C*H*$C\mathit{H}_2\mathrm{CH_3}$); 3.95 (t, 2H, —O$C\mathit{H}_2$—); 6.98 (dd, 2H, Ar—*H*); 8.01 (dd, 2H, Ar—*H*); 11.2 (s, 1H, —COO*H*).

4-(10-undecylen-1-yloxy)biphenyl-4′-[(*s*)-2-methyl-1-butoxy]benzoate ($\mathbf{M_1}$)

Compound **3** (12.5 g, 0.06 mol) was reacted at 65°C with 30 mL of thionyl chloride containing a few drops of DMF for 6 h, and then the excess thionyl chloride was removed under reduced pressure to give 4-[(*S*)-2-methyl-1-butoxy] benzoyl chloride. The acid chloride obtained (11.3, 0.05 mol) was dissolved in 15 mL of chloroform and then added dropwise to a solution of compound **2** (17.6 g, 0.05 mol) in 100 mL of chloroform and 4 mL of pyridine. The mixture was refluxed for 10 h, cooled to room temperature, and then filtered. The filtrate was concentrated. The crude product was precipitated by the addition of ethanol to the mixture and recrystallized from ethanol. A white crystal ($\mathbf{M_1}$) was obtained.

Yield: 71%. mp: 86°C. α_D^{20}: + (toluene). IR (KBr): 3071 (═C—H); 2926, 2851 (—$\mathrm{CH_3}$, —$\mathrm{CH_2}$—); 1751, 1732 (C═O); 1638 (C═C); 1606, 1504 (Ar—); 1250 $\mathrm{cm^{-1}}$ (C—O—C). ANAL. Calcd for $\mathrm{C_{35}H_{42}O_5}$: C, 77.46%; H, 7.80%. Found: C, 77.12%; H, 8.02%. ^{1}H-NMR ($\mathrm{CDCl_3}$, TMS, δ, ppm): 0.93–1.13 (m, 6H, —$C\mathit{H}_3$); 1.27–2.20 [m, 17H, >C*H*$C\mathit{H}_2\mathrm{CH_3}$ and —$(C\mathit{H}_2)_7$—]; 2.32 (t, 2H, —$C\mathit{H}_2\mathrm{COO}$—); 3.92 (t, 2H, —O$C\mathit{H}_2$—); 4.95 (dd, 1H, ^2J = 1.6, $^3J_{\mathrm{cis}}$ = 10.3, $C\mathit{H}_2$═CH—); 5.02 (dd, 1H, ^2J = 1.6, $^3J_{\mathrm{trans}}$ = 17.3, $C\mathit{H}_2$═CH—); 5.72 (m, 1H, $^3J_{\mathrm{cis}}$ = 10.3, $^3J_{\mathrm{trans}}$ = 17.2, ^3J = 6.3, $\mathrm{CH_2}$═C*H*—); 6.95–7.58 (m, 10H, Ar—*H*); 8.02 (q, 2H, ^3J_o = 7.5, Ar—*H*). ^{13}C-NMR ($\mathrm{CDCl_3}$, TMS, δ, ppm): 11.2, 16.6 (2C, methyl—C); 33.4, 30.0, 30.6, 30.6, 30.0, 29.5, 25.8, 33.7, 26.0, 79.3 (10C, methylene—C); 34.0 (1C, tertiary C); 121.5, 128.3, 128.3, 121.5, 131.2, 113.5 (12C, tertiary C in phenyl); 152.5, 133.0, 133.0, 152.5, 122.8, 163.6 (6C, quaternary C in phenyl); 114.6 (1C, $\mathrm{CH_2}$═); 140.0 (1C, ═CH—); 169.3, 164.7 (2C, C═O).

Biphenyl 4,4′-bis(10-undecylen-1-yloxybenzoate) ($\mathbf{M_2}$)

4-(10-Undecylen-1-yloxy)benzoyl chloride (32.4 g, 0.1 mol) was obtained through the reaction of compound **4** with thionyl chloride. Then, a solution of its acid chloride in 10 mL of chloroform was added dropwise to a stirred solution of 4,4′-dihydroxybiphenyl (9.3 g, 0.05 mol) in 100 mL of chloroform and 8 mL of pyridine. The reaction mixture was refluxed for 10 h, cooled to room temperature, and then filtered. The filtrate was concentrated. The product was obtained with the addition of ethanol to the mixture and purified by recrystallization from THF/ethanol (1:1).

Yield: 68%. mp: 135°C. IR (KBr): 3068 (═C—H); 2975, 2852 (—$\mathrm{CH_2}$—); 1754, 1724 (C═O); 1640 (C═O); 1602, 1508 $\mathrm{cm^{-1}}$ (Ar—). ANAL. Calcd for $\mathrm{C_{48}H_{54}O_8}$: C, 75.96%; H, 7.17%. Found: C, 75.78%; H, 7.29%. ^{1}H-NMR ($\mathrm{CDCl_3}$, TMS, δ, ppm): 1.29–1.32 [s, 20H, —$(C\mathit{H}_2)_5$—]; 1.58 (m, 4H, —$C\mathit{H}_2\mathrm{CH_2COO}$—); 2.01 (m, 4H, $\mathrm{CH_2}$═$\mathrm{CH}C\mathit{H}_2$—); 2.29 (t, 4H, —$C\mathit{H}_2\mathrm{COO}$—); 5.00 (dd, 2H, ^2J = 1.7, $^3J_{\mathrm{cis}}$ = 10.2, $C\mathit{H}_2$═CH—); 5.06 (dd, 2H, ^2J = 1.7, $^3J_{\mathrm{trans}}$ = 17.5, $C\mathit{H}_2$═CH—); 5.81 (m, 2H, $^3J_{\mathrm{cis}}$ = 10.2, $^3J_{\mathrm{trans}}$ = 17.4, ^3J = 6.3, $\mathrm{CH_2}$═C*H*—); 7.18—7.59 (m, 12H, Ar—*H*); 8.15 (q, 4H, ^3J_o = 7.3, Ar—*H*). ^{13}C-NMR ($\mathrm{CDCl_3}$, TMS, δ, ppm): 33.6, 29.7, 30.2, 30.2, 29.7, 29.3, 25.4, 32.1 (16C, methylene—C);

TABLE I
Phase-Transition Temperatures of M_1, M_2, and LCE

Sample	Transition temperature (°C); and corresponding enthalpy changes (J/g); $\frac{\text{Heating}}{\text{Cooling}}$
$\mathbf{M_1}$	$\frac{K_1 62.3(1.8)K_2 86.5(23.2)S_C{}^*92.3(2.6)S_A 125.9(4.4)Ch169.3(2.5)I}{I168.5(2.2)Ch124.9(4.4)S_A 91.3(3.4)S_C{}^*81.2(0.4)S_B 52.9(23.5)K}$
$\mathbf{M_2}$	$\frac{K135.6(9.2)N239.4(1.4)I}{I238.2(1.1)N121.5(8.9)K}$
LCE	$\frac{g32.8S_c{}^*115.2(3.1)S_A 232.6(2.9)I}{I225.4(2.3)S_A 111.7(2.7)S_C{}^*28.2g}$

K = solid; g = glassy; S = smectic; Ch = cholesteric; N = nematic; I = isotropic.

121.7, 130.4, 122.5, 128.1 (16C, tertiary C); 157.9, 127.7, 152.2, 132.6 (8C, quaternary C); 114.1 (2C, $CH_2{=}$); 140.2 (2C, $=CH-$); 168.5, 164.2 (4C, $C{=}O$).

Synthesis of the elastomer

LCE was synthesized in 20 mL of freshly distilled toluene, which contained PMHS (1 mmol), $\mathbf{M_1}$ (6.3 mmol), $\mathbf{M_2}$ (0.7 mmol), and 2 mL of a THF solution of H_2PtCl_6 (5 mg/mL). The solution was heated to 65°C under nitrogen and anhydrous conditions and monitored from the Si—H stretch intensity; it went to completion as indicated by IR. LCE was obtained by reprecipitations from a toluene solution into methanol, purified by hot ethanol, and then dried *in vacuo*.

IR (KBr): 2934–2853 ($-CH_3$, $-CH_2-$); 1764, 1736 ($C{=}O$); 1604, 1503 (Ar—); 1300–1000 cm^{-1} (Si—O—Si, C—Si, and C—O—C).

RESULTS AND DISCUSSION

Syntheses

The synthetic routes for the target monomers are shown in Schemes 1 and 2. The structural characterization of the monomers was in good agreement with the prediction. $\mathbf{M_1}$ was synthesized through the reaction of 4-[(*S*)-2-methyl-1-butoxy] benzoyl chloride and compound **2** in chloroform and pyridine. $\mathbf{M_2}$ was obtained through the reaction of 4-(10-undecylen-1-yloxy)benzoyl chloride and 4,4′-dihydroxybiphenyl in the same solvent. IR spectra of $\mathbf{M_1}$ and $\mathbf{M_2}$ showed characteristic bands at 1754–1724, 1640–1638, and 1606–1504 cm^{-1} attributable to ester $C{=}O$, olefinic $C{=}C$, and aromatic $C{=}C$ stretching bands. ^1H-NMR spectra of $\mathbf{M_1}$ showed multiplets at 8.02–6.95, 5.72–4.95, and 3.92–0.93 ppm corresponding to aromatic protons, olefinic protons, and methyl and methylene protons, respectively. ^1H-NMR spectra of $\mathbf{M_2}$ showed multiplets at 8.15–7.18, 5.81–5.00, and 2.29–1.29 ppm corresponding to aromatic protons, olefinic protons, and methylene protons, respectively. LCE was synthesized by a hydrosilylation reaction. The obtained LCE was insoluble in toluene, xylene, DMF, chloroform, and so forth. IR spectra of LCE showed the complete disappearance of the Si—H stretching band at 2166 cm^{-1} and the olefinic $C{=}C$ stretching band. Characteristic Si—O—Si stretching bands appeared at 1300–1000 cm^{-1}. In addition, the ester $C{=}O$ and aromatic absorption bands still existed.

Mesomorphic properties

The mesomorphic properties of $\mathbf{M_1}$, $\mathbf{M_2}$, and LCE were investigated with DSC, POM, and XRD. Their phase-transition temperatures and corresponding enthalpy changes are summarized in Table I. Representative DSC curves of $\mathbf{M_1}$, obtained during the second heating and the first cooling scans, are presented in Figure 1.

Monomer $\mathbf{M_1}$ showed a crystal-to-crystal transition at 62.3°C, a melting transition at 86.5°C, an $S_C{}^*$-to-smectic A (S_A) transition at 92.3°C, an S_A-to-choles-

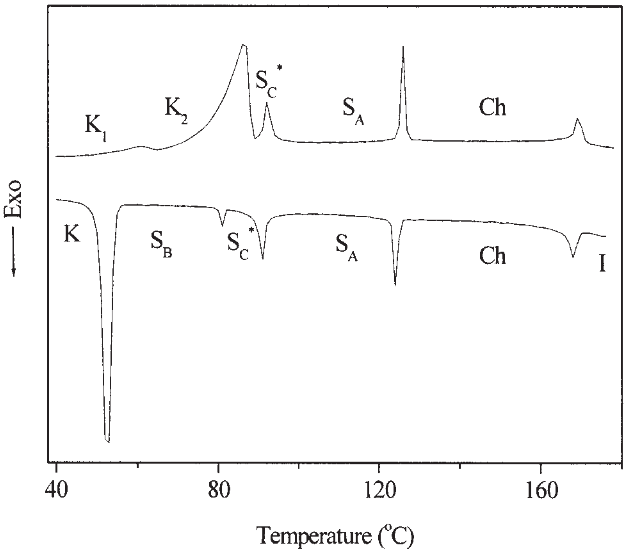


Figure 1 DSC thermograms of $\mathbf{M_1}$.

teric phase transition at 125.9°C, and a cholesteric-to-isotropic phase transition at 169.3°C during heating scans. During cooling scans, an isotropic-to-cholesteric transition at 168.5°C, a cholesteric-to-S_A transition at 124.9°C, an S_A-to-S_C* transition at 91.3°C, an S_C*-to-smectic B (S_B) transition at 81.2°C, and crystallization at 52.9°C were observed. For monomer $\mathbf{M_2}$, a melting transition and a nematic-to-isotropic phase transition appeared at 135.6 and 239.4°C, respectively, and an isotropic-to-nematic phase transition and crystallization temperature appeared at 238.2 and 121.5°C, respectively. DSC heating thermograms of LCE showed a glass transition, an S_C*-to-S_A transition, and an S_A-to-isotropic transition at 32.8, 115.2, and 232.6°C, respectively. The cooling scan showed the same phase transition as the heating scans except for a small supercooling (see Table I).

The optical textures of the LC monomers and elastomer were observed by POM with a hot stage. $\mathbf{M_1}$ exhibited different smectic textures and cholesteric textures, and $\mathbf{M_2}$ exhibited an enantiotropic nematic texture upon heating and cooling. When $\mathbf{M_1}$ was heated to 87°C, the sample began to melt, and mesomorphic properties appeared; a fan-shaped texture of the S_A phase gradually appeared at 96°C, and a cholesteric planar texture appeared at 128°C, which transformed to a cholesteric oily-streak texture with increasing temperature. The textures and mesomorphic properties disappeared at 174°C. During the cooling of the isotropic state, a cholesteric focal-conic texture appeared at 172°C. A fan-shaped texture of the S_A phase appeared at 127°C, a typical fan-shaped texture with a homocentric arc of the S_C* phase appeared at 91°C, and a mosaic texture of the S_B phase appeared at 78°C. Optical textures of $\mathbf{M_1}$ at different temperatures are shown in Figure 2. When $\mathbf{M_2}$ was heated to 134°C, a typical nematic threaded texture appeared, and the texture disappeared at 242°C. When the isotropic state was cooled to 241°C, a nematic droplet appeared; on cooling to 230°C, the nematic threaded texture appeared again and crystallized at 118°C. LCE exhibited the S_A and S_C* fan-shaped textures after annealing for 2 h; this was further confirmed by XRD. A sharp first-order and second-order reflection appeared at a low angle corresponding to the layer spacing, and broad diffusion was found at a wide angle, which was associated with the lateral packing of molecules parallel to one another.

CONCLUSIONS

New LC monomers $\mathbf{M_1}$ and $\mathbf{M_2}$ and a side-chain, chiral LCE were synthesized and characterized. $\mathbf{M_1}$ showed a fan-shaped texture of the S_A phase, a fan-shaped texture with a homocentric arc of the S_C* phase, a mosaic texture of the S_B phase, and cholesteric planar, oily-streak, and focal-conic textures. $\mathbf{M_2}$ showed an enantiotropic nematic thread texture. Besides the S_A phase, chiral LCE also showed an S_C* phase at a moderate temperature and would be a suitable choice for further investigating the piezoelectric effect.

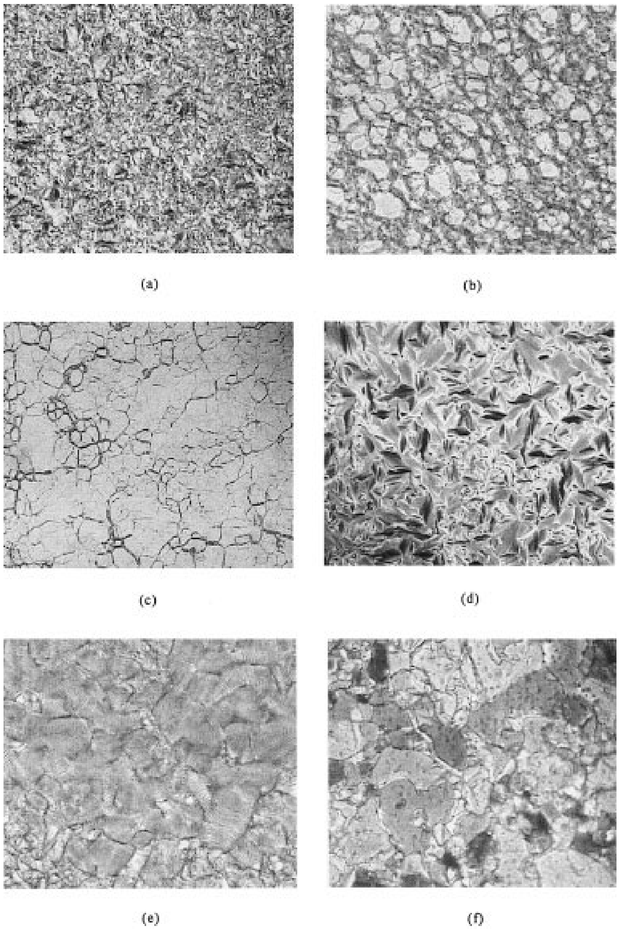


Figure 2 Optical textures of $\mathbf{M_1}$ (200×): (a) fan-shaped texture of the S_A phase on heating to 119.5°C, (b) planar texture of the cholesteric phase on heating to 129.3°C, (c) oily-streak texture of the cholesteric phase on heating to 156.7°C, (d) focal-conic texture of the cholesteric phase on cooling to 166.4°C, (e) fan-shaped texture with a homocentric arc of the S_C* phase on cooling to 88.6°C, and (f) mosaic texture of the S_B phase on cooling to 76.3°C.

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