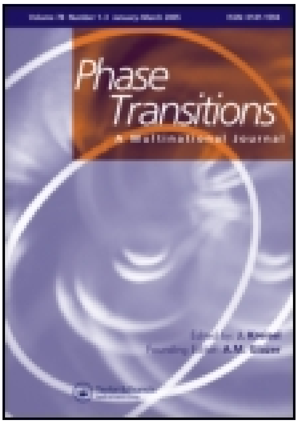


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[a] Department of Organic and Polymeric Materials, Tokyo Institute of Technology, 2-12-1-S8-42 O-okayama, Meguro-ku, Tokyo 152-8552, Japan

[b] School of Physics, University of Hyderabad, Hyderabad 500046, India

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# Anchoring transition in a nematic liquid crystal doped with chiral agents

Tatipamula A. Kumar[ab], Khoa V. Le[a], Satoshi Aya[a], Sungmin Kang[a], Fumito Araoka[a], Ken Ishikawa[a], Surajit Dhara[b] and Hideo Takezoe[a]*

[a]*Department of Organic and Polymeric Materials, Tokyo Institute of Technology, 2-12-1-S8-42 O-okayama, Meguro-ku, Tokyo 152-8552, Japan;* [b]*School of Physics, University of Hyderabad, Hyderabad 500046, India*

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Anchoring transition in cholesteric liquid crystals (CLCs) was studied in this article. The orientational change is not simple in CLCs; instead of the orientational change of the helical axis, a variety of molecular orientations in cells such as planar, Grandjean, fingerprint, and homeotropic orientations were observed depending on chiral dopant, its concentration and temperature. It is proposed that the competition between helix forming power and preferential surface anchoring must be the origin of this phenomenon.

**Keywords:** anchoring transition; cholesteric liquid crystal; chiral dopant; surface interaction



## 1. Introduction

The molecular alignment of liquid crystals (LCs) on treated surfaces is of great importance for the basic understanding of the interfacial phenomena as well as technological applications. The bulk LC and the interfacial properties strongly influence the electro-optic properties of liquid crystal displays (LCDs). Generally, two different categories of alignment layers are used in LCDs, i.e., parallel (homogeneous or planar) and vertical (homeotropic) alignment of the director (the average molecular direction). Apart from LCDs, depending on the strength of the anchoring energy, LCs can also exhibit a rich variety of behaviors such as memory effects, surface melting, orientational wetting, etc.

Recent reports show that the liquid crystal CCN-47 (Merck Japan Ltd., Aikoh-gun, Japan) can show spontaneous discontinuous orientational change (anchoring transition) within the nematic (N) LC phase on amorphous perfluoropolymer (CYTOP, Asahi Glass Co. Ltd., Tokyo, Japan) surface [1]. The same authors also demonstrated a novel bistable memory device writable by a laser beam [2,3] or applying an electric (E) field [3]. The device utilizes the hysteresis of a temperature-driven or an E-field-driven discontinuous anchoring transition in a dye-doped NLC. They also showed that the temperature range of the hysteresis was lowered down to room temperature using a binary mixture system [2]. It is noteworthy that CYTOP provides shock-free alignment layer for smectic LCs [4]. Recently, CYTOP was found to give planar alignment also for NLC mixture ZLI-2293 (Merck); the anchoring energy of ZLI-2293 on CYTOP is almost eighteen times less than

*Corresponding author. Email: takezoe.h.aa@m.titech.ac.jp

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that obtained in polyimide (AL-1254, JSR)-coated cells that are commonly used for planar alignment [5]. It is expected that the low surface anchoring and high transmittance in CYTOP cells will make CYTOP a suitable aligning agent for LCDs as well as many other LC devices.

Cholesteric liquid crystals (CLCs) are chiral analogs of NLCs, and the local director forms a helical structure, which makes this phase more interesting and attractive. It is known that planar and homeotropic alignment layers give helical axes perpendicular and parallel to the surfaces, respectively. However, the situation of surface orientation of molecules is quite different from that in NLCs. Because of the helical structure, the molecular orientation is not compatible to that at surfaces, if surfaces are treated homeotropically. Owing to such a circumstance, a variety of textures emerge in CLCs, i.e., fingerprint, cholesteric finger, Grandjean, and fan-shaped (or focal conic) textures [6]. Then questions arise: (1) Does the helical axis reorient from perpendicular to parallel or remain unchanged, when the favorable surface orientation changes from planar to homeotropic? (2) Does the opposite change in the helical axis occur from parallel to perpendicular upon surface change from homeotropic to planar? (3) Does the complete homeotropic state occur on homeotropic surfaces? If it occurs, it could be a new class of transition; surface-driven cholesteric-N phase transition; (4) Does new type of anchoring transition, e.g., between fingerprint and fan-shaped textures occur? If CCN-47 and CYTOP are used together with chiral dopants, we can address these questions. In this context, the study on the anchoring transition in CLCs provides us additional aspects compared with NLC systems.

From an application viewpoint, CLCs have received considerable attention for applications to optical devices utilizing a spontaneously formed helical structure; one-dimensional photonic structure. In this regard, the switching of a helical axis between normal and tangential to surfaces may provide new applications. For instance, tuning of beam direction in dye-doped CLC lasers becomes possible. Moreover, continuous tuning of wavelength by external stimuli is possible [7,8], if the molecules are released from strongly anchored surfaces, whereas only tuning among discrete wavelengths is possible in a planar-aligned cell configuration with the helical axis oriented perpendicularly to the cell substrates. In this way, the reorientation of helical axis using the anchoring transition is useful from the application viewpoint. Here, we report the anchoring transition in CLCs prepared with several chiral agents.

## 2. Experimental details

We used amorphous perfluoropolymer, poly[perfluoro-(4-vinyloxy-1-butene)] (CYTOP (CTX-809A), Asahi Glass Co., Ltd. Japan), as an alignment layer for this experiment. The CYTOP solution was prepared by dissolving CTX-809A with CT-Solv.180 (Asahi Glass Co., Ltd. Japan) at a weight rate of 1 : 2. Then, it was spin-coated on ITO-coated glass plates using a spin coater. After spin coating, substrates were kept in an oven for curing at 100°C for half an hour. In order to maintain uniform cell thickness, we used glass beads of 5 μm as spacers. These spacers were mixed with glue and a line of glue was applied at the edge of the substrates. Then ultraviolet (UV) polymerization was done by exposing substrates to UV light. CYTOP-coated unrubbed cells were mainly used to observe textures. On the other hand, polyimide-coated wedge cells were used to measure the pitch of the cholesteric liquid crystals. The liquid crystal CCN-47 exhibits the following sequence of phase transitions: Cr 25.6°C SmA 28.2°C N 57.3°C Iso, and has a large

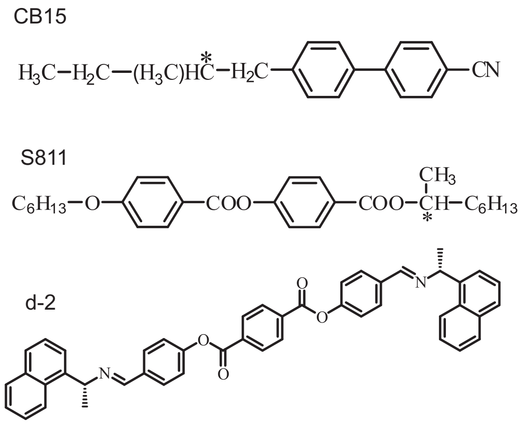


Figure 1. Chemical structures of chiral agents CB 15, S811, and d-2.

negative dielectric anisotropy ($\Delta\varepsilon = -5.7$ at 30°C), where Cr, SmA, and Iso stand for crystal, smectic A, and isotropic phases.

We used several chiral agents, i.e., 4-cyano-4′-(2-methylbutyl)-biphenyl (CB15, Merck), 4-[1-methylheptyloxy]carbonylphenyl-4-(hexyloxy)benzoate (S811, Merck) and an imine-based chiral dopant (d-2) [9]. CB15 has a phase sequence Cr 4°C N* 54°C Iso, and exhibits right-handed helix. S811 has a phase sequence Cr 47.8°C Iso, and exhibits left-handed helix. The chemical structures of CB15, S811 and d-2 chiral agents are shown in Figure 1. We prepared several CLCs by adding chiral agents of various concentrations to CCN-47. The texture observation was made using a polarizing optical microscope (POM). The temperature of the sample was controlled by a Mettler hot stage (FP82HT).

## 3. Results and discussion

### 3.1. *CLCs doped with CB15*

We first prepared a CLC by adding 5 wt% of CB15 to CCN-47. Then it was injected into the CYTOP-coated unrubbed cell of thickness ~5 μm. When the sample was cooled from Iso to cholesteric phase at a rate of 1°C per minute, the fan-shaped texture appeared at 51.1°C as shown in Figure 2(a). A closer look at Figure 2(a) indicates the existence of fringe structures (see inset). When the temperature reached 48.0°C, the fan-shaped texture changed to a fingerprint texture as shown in Figure 2(b)–(d). We also observed the sample on heating and found that the texture changed from fingerprint to fan-shaped at 48.3°C as shown in Figure 2(e) and (f). Thus, the anchoring transition between fingerprint and fan-shaped textures occurs. Interestingly, however, we did not observe any anchoring transition to a complete homeotropic orientation in this concentration. This suggests that the interfacial forces that are responsible for a conventional anchoring transition are affected by the addition of chiral dopant. In other words, helix-forming ability is strong enough compared with the interfacial forces.

To reduce the helix-forming ability, we reduced the concentration of CB15 from 5 to 3 wt%. The textures are shown in Figure 3. When the temperature was reduced to 55.4°C from the Iso phase, only a planar state existed with several cholesteric defects in it as shown in Figure 3(a). As the temperature decreased further to 46.7°C, the fingerprint texture disappeared and some regions with reduced birefringence were seen, as shown in Figure 3(b), suggesting a tilting of the director. This transition proceeded until it

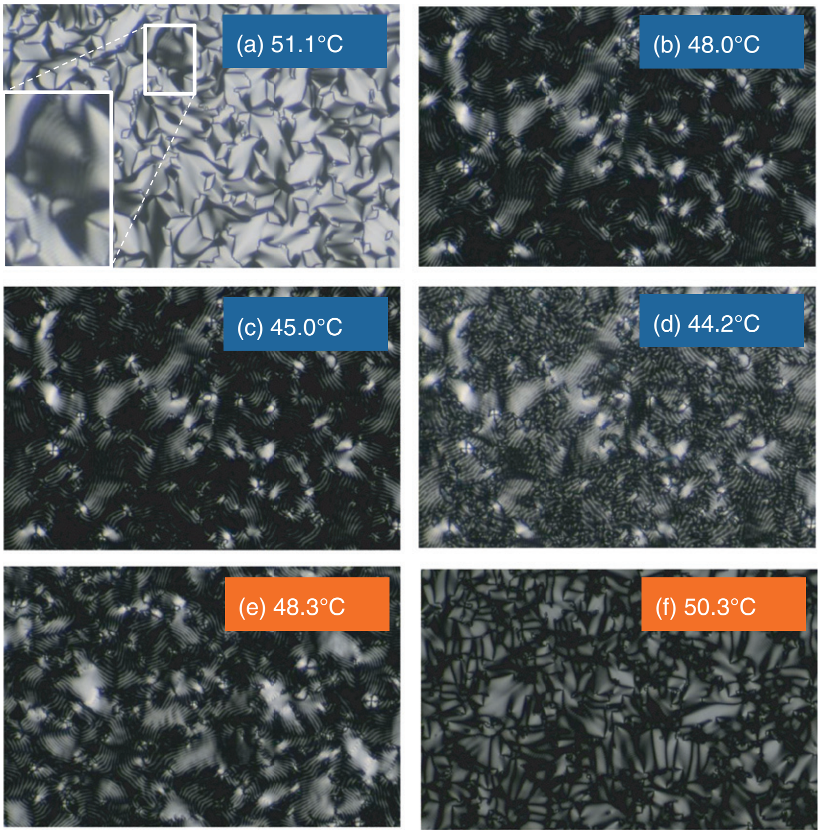


Figure 2. The evolution of textures in CCN-47 doped with 5 wt% of CB15: (a) fan-shaped texture, (b)–(d) fan-shaped to fingerprint on cooling, and (e) and (f) fingerprint to fan-shaped on heating. Even in (a) fine fringes are seen (inset). Polarizer and analyzer directions are horizontal and vertical. Blue and orange labels for temperatures are those on cooling and heating, respectively.

reached 45.7°C. On further cooling, some small dark regions were formed at 39.7°C and became more prominent when the sample was cooled to 28.7°C as shown in Figure 3(c). Almost complete homeotropic state with some thin bright lines were observed when the temperature was reduced to 25°C, as shown in Figure 3(d). On heating, the cholesteric fingers appeared from homeotropic domains at 27.3°C, as shown in Figure 4(a), and the texture remained the same until the temperature reached 51.3°C as shown in Figure 4(b). At 51.4°C, some bright domains were also seen along with the fringe texture as shown in Figure 4(c) and (d). Gradually the fringe texture disappeared as the temperature proceeded towards Iso as shown in Figure 4(e) and (f). The situation is clearer in the sample with 1 wt% of CB15. By decreasing temperature, fringe textures could not be observed. Instead, the anchoring transition could be more clearly observed. Thus, with decreasing the weight fraction of chiral dopant CB15, the anchoring transition becomes prominent, as expected.

### 3.2. *CLCs doped with S811*

We further studied a cholesteric liquid crystal prepared by mixing the chiral agent S811 with CCN-47. The behavior in the sample with 5 wt% of S811 was similar to that in the samples with 5 wt% of CB15, although the anchoring transition from fan-shaped to

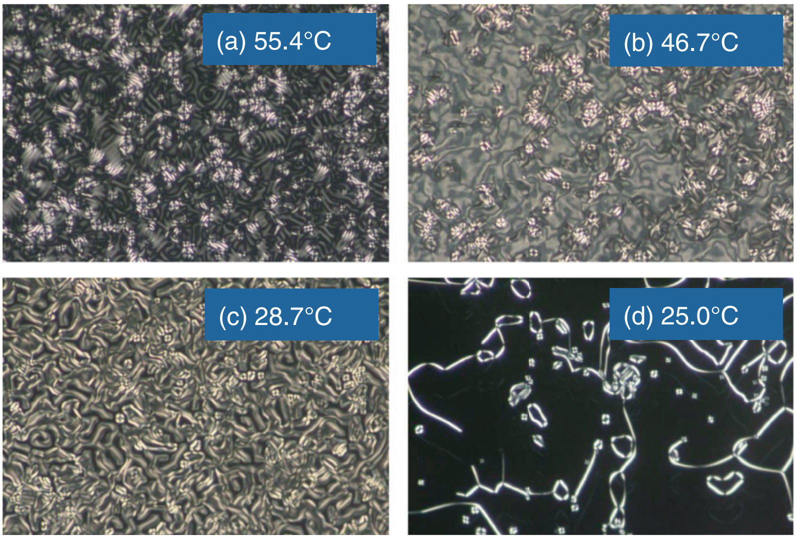


Figure 3. Photomicrographs in CCN-47 doped with 3 wt% of CB15 on cooling: (a) planar state with some cholesteric defects, (b) planar to tilt transition, (c) fingerprint texture with some homeotropic regions, and (d) homeotropic state with a few thin bright lines. Polarizer and analyzer directions are horizontal and vertical.

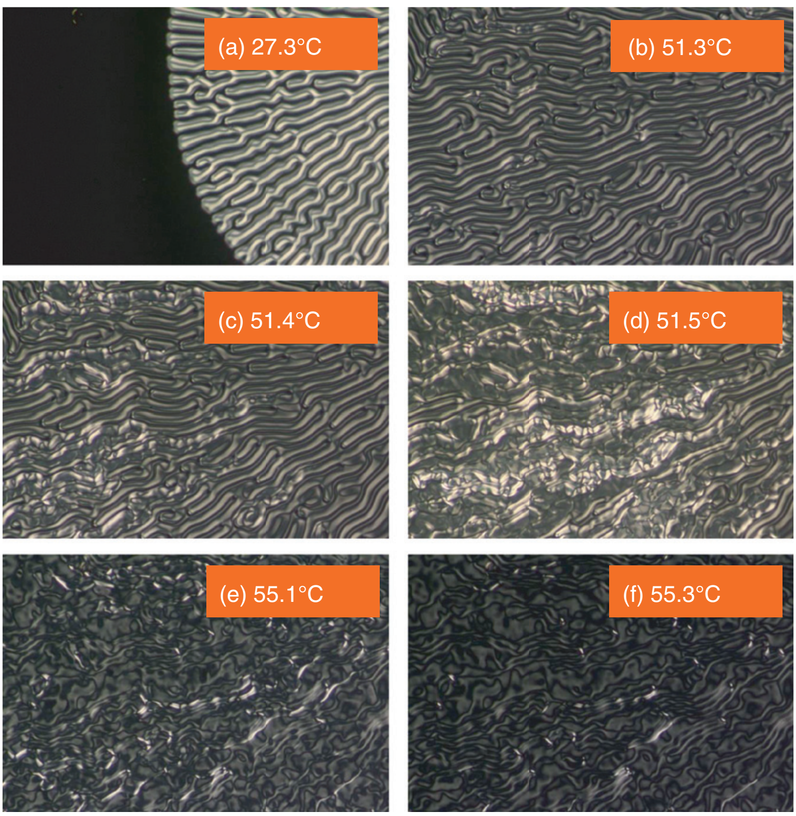


Figure 4. Photomicrographs in CCN-47 doped with 3 wt% of CB15 on heating: (a) the transition from homeotropic to fingerprint, (b) fingerprint texture, (c) and (d) the transition from fingerprint to tilted state, and (e) and (f) tilted state with reduced brightness. Polarizer and analyzer directions are horizontal and vertical.

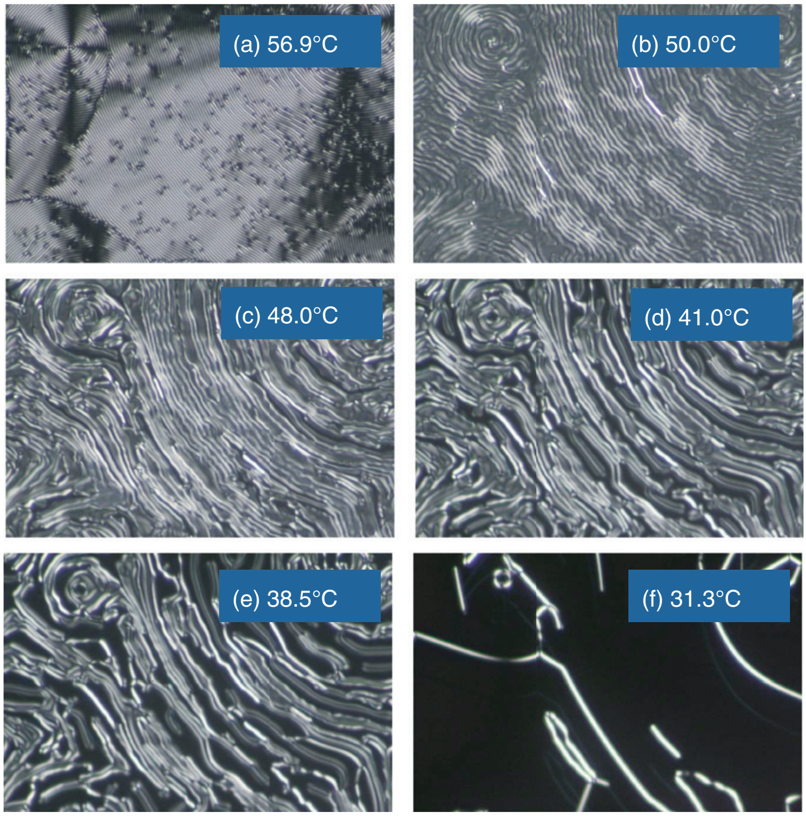


Figure 5. Photomicrographs in CCN-47 doped with 1 wt% of S811 on cooling: (a) fan-shaped texture with fringes, (b)–(d) the gradual transition from fan-shaped to fingerprint, and (e) and (f) the transition from fingerprint to homeotropic state. Polarizer and analyzer directions are horizontal and vertical.

fingerprint at ~31.5°C and to homeotropic at ~30°C was observed in the former sample. Here we only show the behavior of the sample with 1 wt% of S811. Mostly the fan-shaped texture with fringe texture was observed during the Iso to cholesteric phase transition as shown in Figure 5(a). As the temperature decreased a fingerprint texture was gradually transformed to a homeotropic texture as shown in Figure 5(b)–(e). When the sample temperature was reduced further, the fingerprint texture almost disappeared and became nearly homeotropic as shown in Figure 5(f).

The variation is more clearly seen when the same sample (1 wt%) was filled in CYTOP-coated rubbed cell. With decreasing temperature from Iso to cholesteric, a tilted state appeared first as shown in Figure 6(a). The overall brightness of the tilted state increased as the temperature decreased. At around 37.0–35.0°C the tilted state changed to a planar state as shown in Figure 6(b). When the temperature reached 32.0°C, the anchoring transition from planar to homeotropic state occurred along the rubbing direction as shown in Figure 6(c). Here the anchoring transition at both surfaces normally occurred separately, as indicated by three different brightnesses. With further decreasing temperature the homeotropic regions grew as shown in Figure 6(d) and (e), and became completely homeotropic at 30°C as shown in Figure 6(f). On the other hand, the sample exhibited homeotropic state on heating till the temperature reached 37.8°C as shown

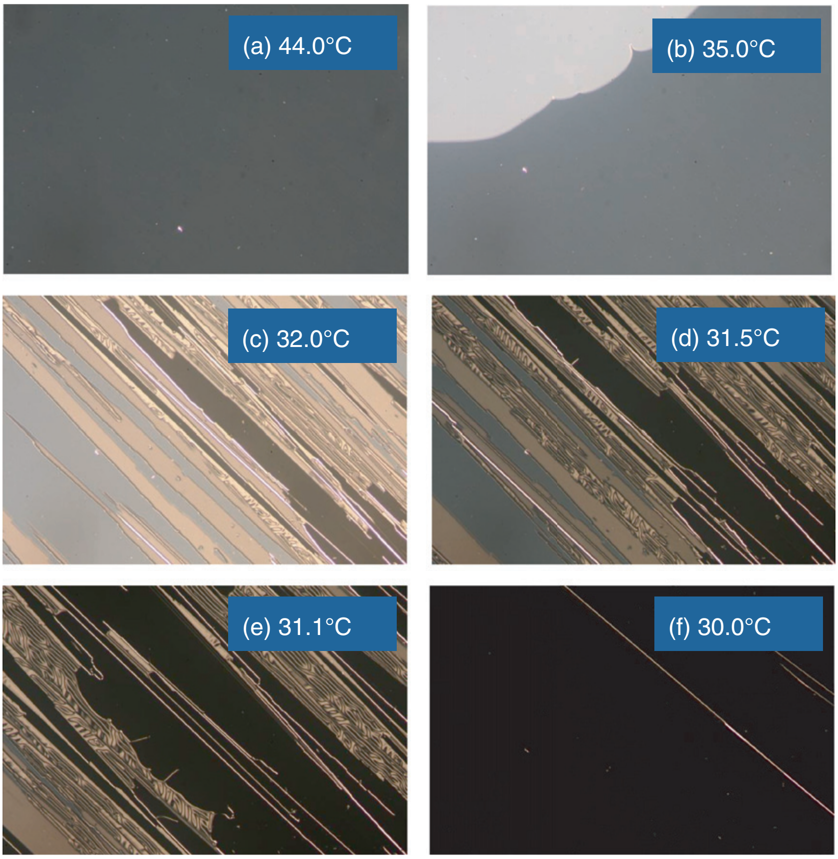


Figure 6. Photomicrographs in CCN-47 doped with 1 wt% of S811 in a CYTOP-coated rubbed cell on cooling: (a) tilted state, (b) tilted state with increasing brightness, (c)–(e) anchoring transition from planar to homeotropic, and (f) homeotropic sample. Polarizer and analyzer directions are horizontal and vertical.

in Figure 7(a). When the temperature reached 38°C, it exhibited anchoring transition from homeotropic to planar state as shown in Figure 7(b) and (c). As the temperature approached CLC-N transition, it exhibited a tilted state with reduced brightness as shown in Figure 7(d).

### 3.3. *CLCs doped with d-2*

The two chiral dopants CB15 and S811 have similar helical twisting power (HTP) [10,11]. In order to study the effect of the dopant with large twisting power, we chose d-2, which is known to have a high helical twisting power ($\mathrm{HTP} = 140\,\mu\mathrm{m}^{-1}$ in the host 6OCB) [9]. We first prepared a CLC by adding 5 wt% of d-2 with CCN-47 and it was introduced in a CYTOP-coated unrubbed cell. When the temperature decreased from the Iso phase, we found Iso to cholesteric phase transition at 58.7°C as shown in Figure 8(a). As the temperature decreased, a dark bluish color due to selective reflection was observed with Grandjean steps as shown in Figure 8(b). When the temperature decreased further, the color shifted from blue to cyan, and the width of Grandjean steps also increased as shown in Figure 8(c) and (d). With this concentration we did not see the anchoring

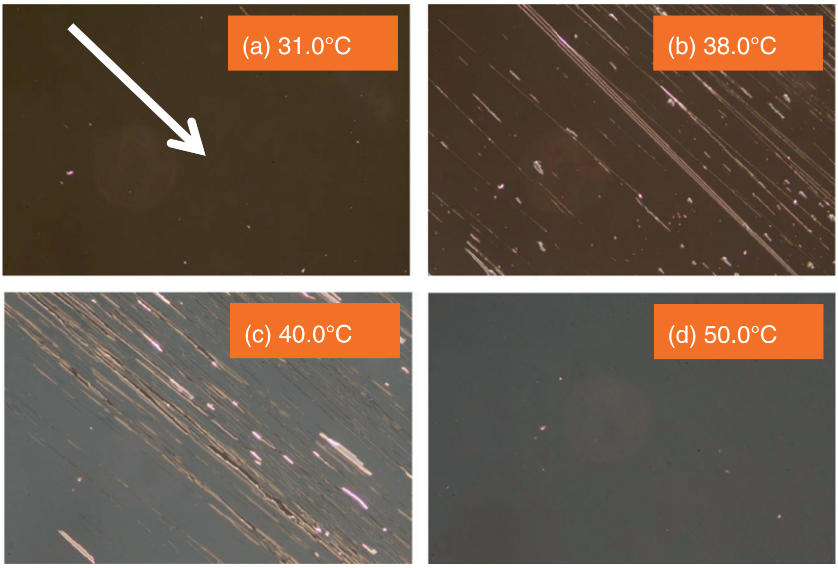


Figure 7. Photomicrographs in CCN-47 doped with 1 wt% of S811 in a CYTOP-coated rubbed cell on heating: (a) homeotropic state at 31.0°C, (b) and (c) anchoring transition from homeotropic to planar state, and (d) tilted state with reduced brightness. Polarizer and analyzer directions are horizontal and vertical, unless otherwise stated.

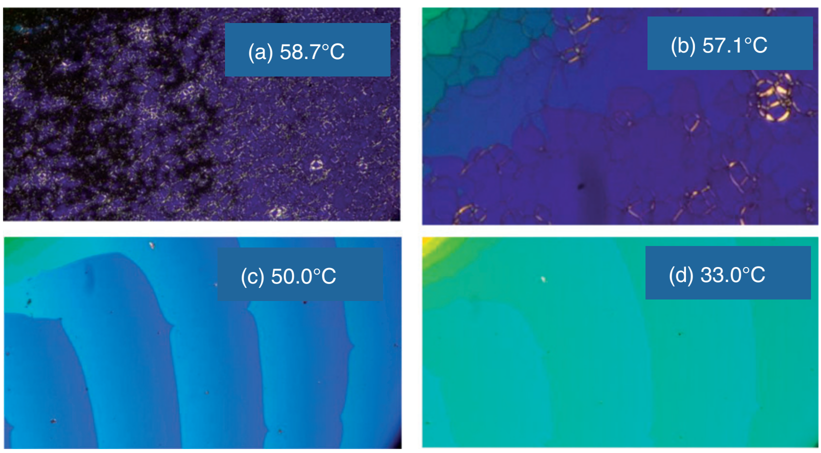


Figure 8. Evolution of textures with selective reflection on cooling in CYTOP-coated rubbed cell with 5 wt% of d-2: (a) Iso to cholesteric phase transition, (b) selective reflection with Grandjean steps, (c) selective reflection of blue color, and (d) selective reflection of cyan color. Polarizer and analyzer directions are horizontal and vertical.

transition in the sample. No anchoring transition was observed in a sample with 0.7 wt% of d-2 either. Then we reduced the concentration of d-2 to 0.5 wt% and made microscopic observations as shown in Figure 9. As the temperature decreased from Iso phase, we observed focal conic texture with fingerprints at 58.5°C as shown

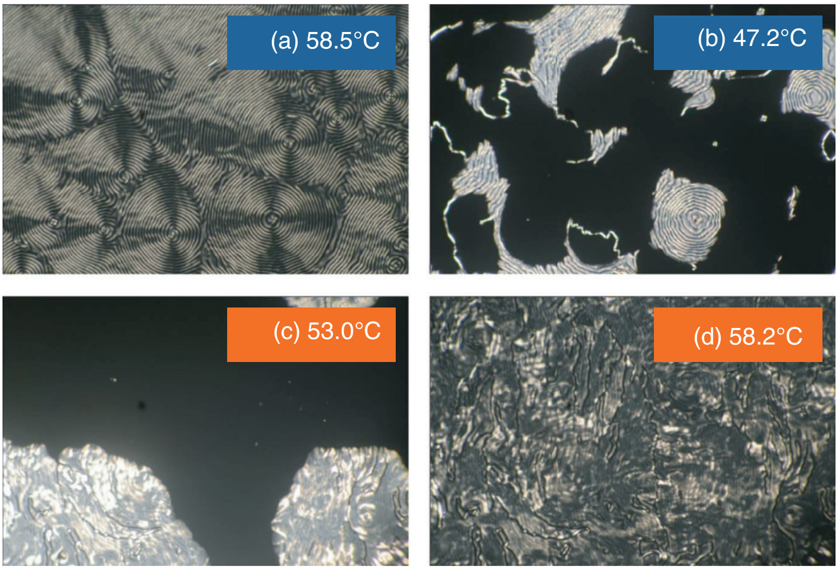


Figure 9. Photomicrographs in CCN-47 with 0.5wt% of d-2: (a) focal conic texture, (b) discontinuous anchoring transition from planar to homeotropic on cooling, (c) discontinuous anchoring transition from homeotropic to planar on heating, and (d) tilted state. Polarizer and analyzer directions are horizontal and vertical. Blue and orange labels for temperatures are those on cooling and heating, respectively.

in Figure 9(a). We also observed a discontinuous anchoring transition from tilted to homeotropic state at 47.2°C as shown in Figure 9(b). On heating it exhibited anchoring transition from homeotropic to tilted state at temperature 53.0°C as shown in Figure 9(c) and (d).

We also prepared a contact cell of CLC and NLC (pure CCN-47), and observed the texture to investigate how much concentration of chiral dopant is needed to have a uniform helix with the helical axis along the cell surface normal. For this experiment we used CYTOP-coated rubbed cells and prepared CLC by doping CCN-47 with 0.7 wt% of d-2. We filled the cell with pure CCN-47 and CLC in their Iso phase. As shown in Figure 10(a) and (b), the texture exhibited Grandjean steps as the temperature was decreased from Iso. A texture under decrossed polarizers is also shown in Figure 10(c). It exhibited Grandjean steps till it reached 32.2°C, at which we observed discontinuous anchoring transition on the CCN-47 side and continuous anchoring transition on the CLC side. While the continuous anchoring transition took place, we also observed fingerprint texture as shown in Figure 10(d). A similar behavior was observed on heating as shown in Figure 10(e) and (f).

We also measured the pitch of the mixture with concentrations of 7.5 wt%, 5 wt% and 0.7 wt% of d-2. We used polyimide-coated wedge cells with a wedge angle $\alpha = 1.54^\circ$. In order to measure a pitch, we took several wedge lines and measured the pitch below $\sim$10°C from the Iso-cholesteric phase transition temperature. The pitch of the CLC was measured using the following equation.

$$P = 2s\tan\alpha,$$

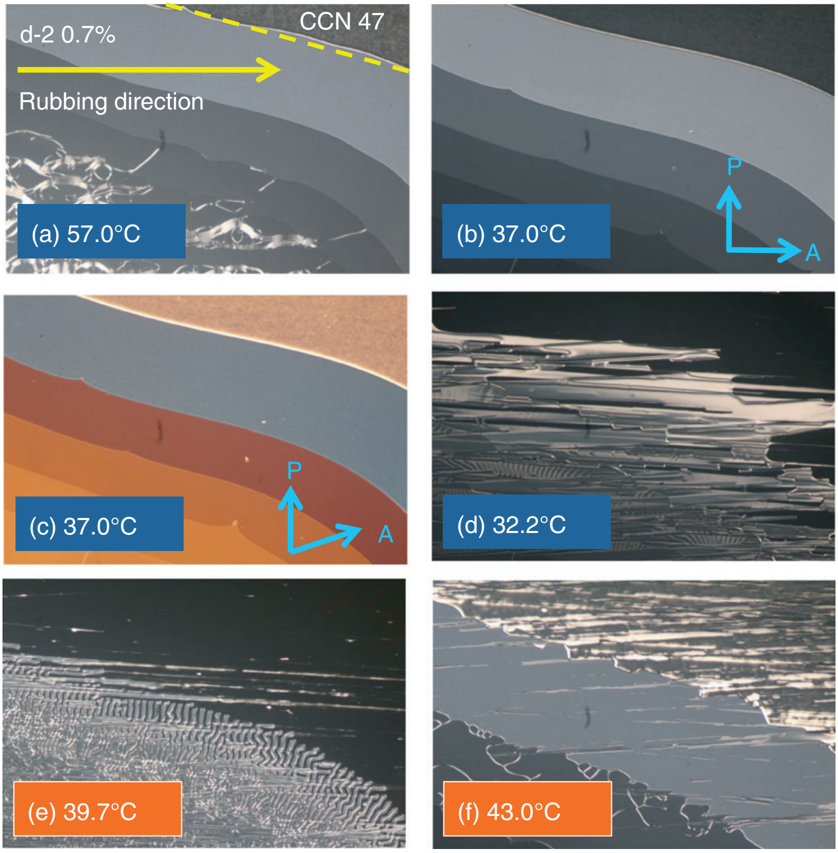


Figure 10. Photomicrographs in a contact cell of pure CCN-47 and CCN-47 with 0.7 wt% of d-2: (a) and (b) Grandjean steps observed with decreasing temperature from the Iso phase, (c) the same as in (b) under slightly decrossed polarizers, (d) discontinuous (NLC side) and continuous (CLC side) anchoring transition, (e) fingerprint texture emerging during the anchoring transition on heating, and (f) after anchoring transition on heating. Polarizer and analyzer directions are horizontal and vertical, unless otherwise stated. Blue and orange labels for temperatures are those on cooling and heating, respectively.



where $s$ is the distance between the wedge lines. The temperature dependence of the pitch obtained is shown in Figure 11(a). The pitch was $\sim$0.54 μm at a temperature very close to the Iso to cholesteric phase transition, increasing to 0.59 μm near room temperature 30°C. In the same temperature range, the pitch exhibited similar temperature dependence from 4.4 to 5.2 μm in the sample with 0.7 wt% of d-2. We also estimated the HTP of d-2. The HTP of a chiral dopant is defined as

$$\beta = (Pcr)^{-1},$$

where $P$ is the pitch of CLC, $c$ is the molar fraction of the dopant, and $r$ is the enantiometric excess of the dopant. The variation of the inverse of the pitch as a function of molar fraction of d-2 is plotted in Figure 11(b). HTP was found to be $\beta \sim 70\,\mu\text{m}^{-1}$, which was almost a half compared with the value in the host liquid crystal 6OCB [9]. Table 1 is a summary of the result on various chiral dopants.

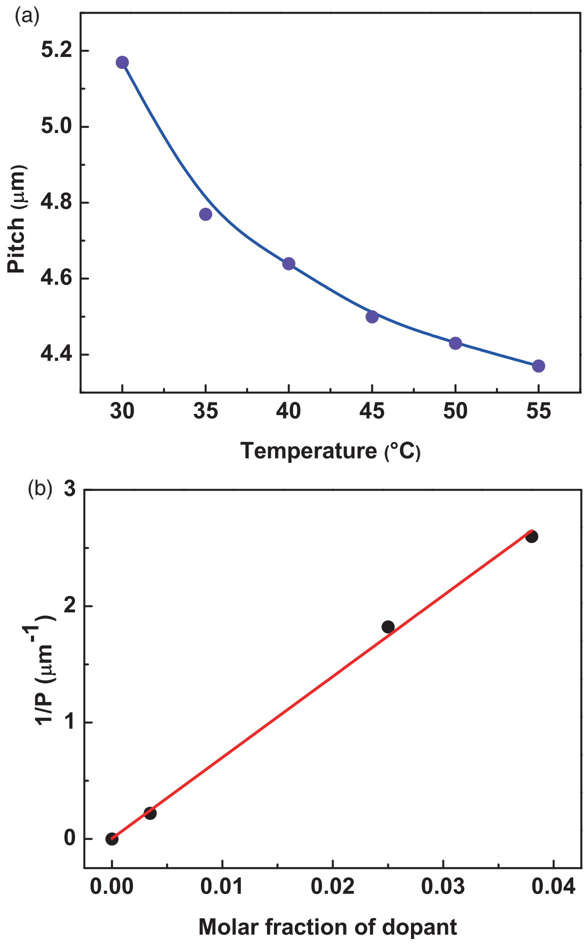


Figure 11. (a) Variation of pitch as a function of temperature for the concentration 0.7 wt% of d-2. (b) Variation of inverse pitch as a function of molar fraction of dopant.

Table 1. Summary of Iso to N and anchoring transition temperatures in CCN-47 with chiral dopants.

| Chiral dopant (wt%) | Iso-N (°C) | Anch. Trans. (°C on cooling) | Anch. Trans. (°C on heating) |
|---|---|---|---|
| None | 57.9 | 47.8 (P to H) | 54.0 (H to P) |
| CB15 (5 wt%) | 51.4 | ~44 (Fan to Fin) | ~48 (Fin to Fan) |
| CB15 (3 wt%) | 55.4 | ~47 (Fan to T) | ~27 (H to Fin) |
| | | ~30 (T to H) | 51.4 (Fin to T) |
| S811 (5 wt%) | 55.1 | ~38 (Fan to Fin) | ~32 (H to Fin) |
| | | ~32 (Fin to H) | ~52 (Fin to Fan) |
| S811 (1 wt%) | 56.9 | ~48 (Fan to Fin) | ~38 (H to Fan) |
| | | ~38 (Fin to H) | |
| d-2 (5 wt%) | 58.9 | no ATr | no ATr |
| d-2 (0.7 wt%) | 58.5 | no ATr | no ATr |
| d-2 (0.5 wt%) | 58.5 | ~47 (Fin to H) | ~53 (H to Fin) |

Note: P (planar), H (homeotropic), Fan (fan-shaped), Fin (fingerprint), T (tilted).

## 4. Conclusions

We have observed the anchoring transitions in CLCs prepared by several chiral agents such as CB15, S811, and d-2. Very complicated anchoring transitions were observed mainly depending on HTP. When HTP was very large, a helical structure with the axis perpendicular to surfaces appeared with clear selective reflection and Grandjean lines, and no anchoring transition was observed. In smaller HTP samples, on cooling, a fan-shaped texture with fine fringes was transformed to a fingerprint texture and further to a homeotropic texture. On heating, a cholesteric finger emerged from the homeotropic texture, and then the anchoring transition to fingerprint and to fan-shaped texture occurred. Such complicated anchoring transition originates from the competition between surface interaction and helix-forming power.



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