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The investigation of glass-forming liquid crystalline materials for mid wave infrared polarization applications is driven by their low melt viscosity and ability to vitrify order, and thus functionality, into films with a wide range of thicknesses. Commercially available polarizers that function in the mid-wave infrared region suffer from poor polarization contrast, high cost, and limited size. In this work, we explore the feasibility of using cyclic siloxane-based liquid crystalline materials with chiral mesogens to form circular polarizers in the mid-wave infrared spectrum (3–5 μm). Specifically, we have designed a cholesteric molecular blend to possess the proper helical twisting power to exhibit a selective reflection notch in the 4.0–4.5 μm region. We have fabricated circular polarizers using shear under a number of processing conditions, and explored their performance as measured by polarization contrast. Processed films with reflection notches at the proper wavelength and near theoretical reflection performance have been prepared. A proper balance between film thickness and cooling rate has led to the demonstration of optically transparent and uniform films on 1 inch diameter substrates. The use of simple alignment layers was demonstrated to yield consistent formation of Grandjean monodomains, by reducing the tendency of the large pitch blends to spontaneously form a fingerprint molecular orientation observed in cells with untreated surfaces. The measured polarization contrast of >70:1 exceeds the values obtained from state-of-the art commercial polarizers in this wavelength region.

1. Introduction

The use of linear polarizers in the visible part of the spectrum is widespread. Inexpensive, versatile, high-performance thin-film polarizers of many shapes and sizes are commercially available. This availability is critical to the performance of most electro-optic technologies that use polarizer/analyser technology as a discriminant in information transfer. This is not the case in the mid-wave ($\lambda = 3$–5 μm) and long-wave ($\lambda = 8$–11 μm) infrared (MWIR and LWIR) spectral bands, however, as few thin-film polarization technologies exist for these regions. Existing polarizers for these regions are mainly 'wire-grid' type elements consisting of regularly spaced metallic wires on an IR-transparent substrate. For these systems to operate efficiently, the grid spacing (d) must

be much less than half the design wavelength ($d \ll \lambda/2$). This requirement causes fabrication to become more and more challenging as the wavelength of interest decreases from the LWIR to MWIR. In addition to being expensive and only available in small area devices ($A = 1$–2 in^2), the main limitation of these polarizers in the midwave MWIR is their available contrast ratio. This measure of performance is defined as the transmission efficiency for linearly polarized light whose electric field vector is perpendicular to the wire grid direction relative to the intensity whose electric field is parallel to the wire direction. Although respectable contrast ratios are obtainable in the LWIR (>150:1 at 10 μm), only limited contrast ratios (<25:1) are available in commercial polarizers for the MWIR spectral band. As a comparison, typical linear polarizers used in the visible spectral band have a contrast ratio greater than 5000:1.

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Cholesteric liquid crystals (LCs) offer a convenient alternative method of polarization. The selective reflection properties, helical twisting power, and molecular variables affecting these materials have been the basis of a large amount of research during the past two decades [1, 2]. This class of material is well known to function as a spectrally selective circular polarizer; i.e. transmitting one handedness of radiation through a thin film while completely reflecting the other handedness. A cholesteric material possessing a right-handed twist will selectively reflect all right-handed radiation at a wavelength governed by the well known relationship

$$\lambda = nP. \qquad (1)$$

Here, n is the average refractive index and P is the pitch of the compound (distance taken for the director to rotate 360°). The other handedness is transmitted at this wavelength and thus the film acts to polarize initially unpolarized light. Liquid crystal circular polarizers have advantages over other polarization schemes. They are simple single-element devices, exhibit no absorptive losses, possess excellent optical quality and have very high damage thresholds [3]. Materials with reflection properties from the UV to the LWIR can be formed by properly adjusting the pitch of the material. Compounds with short pitches result in UV or visible reflecting materials, while longer pitches result in reflection somewhere in the IR region. To date, most work on cholesteric materials has been for applications which utilize selective reflection in the visible part (400–650 nm) of the spectrum. When macroscopically aligned into Grandjean (planar) monodomains, these films exhibit bright colours owing to selective reflection of a particular wavelength. Schemes to control the bandwidth have also been demonstrated, typically by spatially grading the pitch within a film [4–6].

In order to make solid thin-film cholesteric-based polarizers, the candidate material system must possess the capability to be vitrified into a solid while maintaining its inherent LC properties. Such 'frozen' LC systems are currently of interest for a number of applications. Main-chain, side-chain, and networks of cholesteric meso-phases are all candidate materials systems, each with particular advantages and disadvantages [7, 8]. The largest detriment to these material classes is the inherently large melt viscosity which hinders good alignment and sub-stantially increases annealing times. Even in optimum conditions, large monodomains are typically difficult to achieve. In an alternative class of glass-forming materials, called excluded volume liquid crystals, suitable for this application, LC mesogens are tethered onto small cores to allow relatively low molecular mass materials (and thus much lower melt viscosities) to be formed. Hinderance of packing of the mesogens by steric crowding

suppresses crystallization and facilitates the formation of a low temperature glassy phase. Thus, it is possible to process the material while in the mesophase, obtain the correct macroscopic order, and then cool or quench the sample to room temperature where the order is frozen into a stable glass. Chen *et al.* [9–11] have pioneered the structure–property investigation of a wide range of carbon-based cores, examining core geometry and mesogen effects on the stability and propensity to form stable glass-forming LC films. Another subset of this class of materials examined extensively in the past is based on cyclic-siloxane based cores with attached mesogens [12–14]. Considerable work has been done to examine the cholesteric properties of a wide variety of structures, as these compounds have been used for many optical applications. To date however, all of the work has centred on controlling the reflection band in the visible and near IR (NIR) part of the spectrum. No work has been published to date on utilizing their ability to form stable reflective glasses for application in the MWIR.

The work presented here examines the feasibility of forming high-contrast, MWIR polarizers from these cyclic-siloxane based cholesteric materials. The LC macromolecule consists of cholesteryl-4-allyloxybenzoate (C4AB) and biphenyl-4-allyloxybenzoate (B4AB) meso-gens attached to a pentasiloxane ring [14, 15]. By controlling the relative amount of C4AB to B4AB, the helical twisting power was varied. Previously synthesized materials [15] were blended to yield a compound exhibit-ing a selective reflection notch at approximately 4.0 μm. This material was shear-processed in the mesophase and subsequently quenched to room temperature to form a stable, ordered cholesteric glass. Various processing con-ditions including film thickness, cooling rate, shearing temperature, and substrate type were investigated. The use of a polyimide buffing layer to increase the morphological homogeneity of the coating was also explored. The work presented indicates that it is feasible to achieve high-contrast MWIR polarizers with comparable performance to state-of-the-art commercially available devices using this class of materials.

2. Experimental procedures

The molecular structure of the mesogens and macro-molecule are shown in figure 1. Previous work has evaluated the selective wavelength versus chiral content over a narrow range of compositions for this class of compounds [14]. Blending of compounds possessing 0 and 15 mol % cholesterol-containing macromolecules was found to be the best method for preparation of materials with the proper helical twisting power. Both of these compounds have been synthesized and charac-terized previously [15]. Blends were formed by mixing
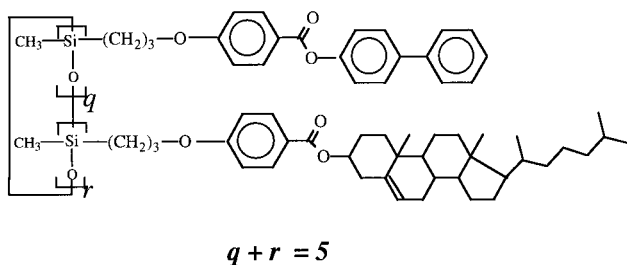
$q + r = 5$

Figure 1. Chemical structure of cholesteric cyclic-siloxane liquid crystalline materials. Compound 1 is a blend of a compound with $r/q = 0$ and a compound with a $r/q = 0.176$ to yield compound 1 which has roughly 4.5 mol % cholesterol. Compound 2 has a $r/q = 1$.

the proper amounts of each compound in toluene overnight, followed by standard precipitation into methanol. Homogeneity was checked using polarized optical microscopy of films prepared from the resulting powder. Macromolecules containing 50 mol % cholesterol (synthesized previously and not blended) were investigated for comparison purposes.

Thermal analyses were conducted on second heating and first cooling scans using a Thermal Analyst TA-MDSC and a $10°C \, min^{-1}$ heating rate in a standard mode. A Nikon Optiphot-Pol microscope, a Mettler FP90 Central Processor, and a Mettler FP82HT hot stage were used for polarizing optical microscopy (POM) to examine liquid crystal textures and conduct cooling and annealing experiments. FTIR examination was performed on a Perkin-Elmer 1725X spectrometer.

The polarization contrast, or extinction ratio, was measured using an experimental set-up shown schematically in figure 2. This system is capable of measuring the spectrally dependent polarization characteristics of an optical device. The combination of a variable filter wheel, linear polarizer, and a variable waveplate enables one to generate circularly polarized light (with either right- or left-handedness) that can be directed toward the film. The waveplate converts linear polarized light into circularly polarized light. The amount of light transmitted through the system, with and without the sample present, was measured by the detector. Transmission was measured for both left- and right-handed polarization states at wavelengths corresponding to the selective reflection wavelength.

The two kinds of IR transparent substrates onto which the polarizers were shear-processed were CaF_2 (Aldrich) and sapphire. Films were typically heated into the isotropic phase, cooled down to 150°C and sheared by translation of the opposing substrate. The films were cooled with prescribed cooling rates to room temperature. Single substrate films were formed by using a sapphire or CaF_2 bottom substrate and a NaCl plate as the top substrate. For the preparation of single substrate films, the NaCl plate was subsequently dissolved in warm water. Spacers were used to control sample thickness and consisted of chopped glass fiber (15 μm) or kapton film (75 μm). For alignment layer studies, a polyimide precursor solution (Aldrich, 2% in NMP) was used to coat appropriate substrates, using a commercial spin coater at 3000 rpm, and then baked in air at 100°C for 24 h. POM measurements were performed on standard laboratory glass slides.

3. Results and discussion

The phase diagram for the siloxane core/mixed mesogen class of macromolecules [12] indicates that the 0% cholesterol compound is nematic while the 15% cholesterol compound exhibits a cholesteric mesophase with a reflection band near 1050 nm. Based on extrapolation of data in previous work [14], the proper concentration to achieve a notch in the 4–4.5 μm ($2500-2222 \, cm^{-1}$) regime of the MWIR is approximately 4.5 mol % cholesterol. Therefore, a blend was formed by mixing (in solution) the macromolecule possessing 0% and 15% in the proper proportions. This blend will be referred to as compound 1 while the macromolecular possessing 50% cholesterol will be referred to as compound 2. The DSC cooling data of compound 1 is shown in figure 3. It possesses a broad cholesteric region ranging from the I–Ch transition at 189°C to the glass transition temperature at approximately 62°C, upon cooling. The cholesteric nature of the compound was confirmed using POM. The thermal transitions of compound 2 differ only slightly with $T_{cl} = 220°C$ and a $T_g = 50°C$ being measured previously [15]. The selective reflection wavelength of compound 2 is 480 nm.

Initial attempts at forming circular polarizers in the MWIR region indicated that strong notches could be developed at the target wavelength for both CaF_2 and
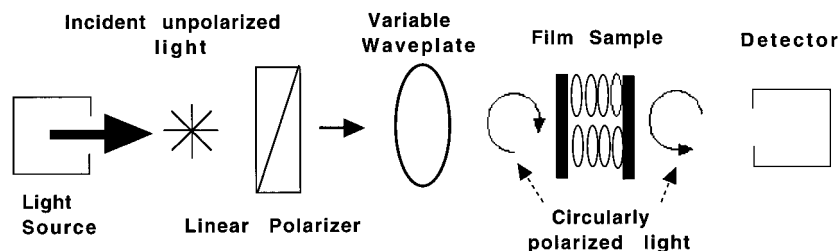
Figure 2. Schematic diagram of optical set-up to determine the polarization contrast of processed films in the MWIR region.
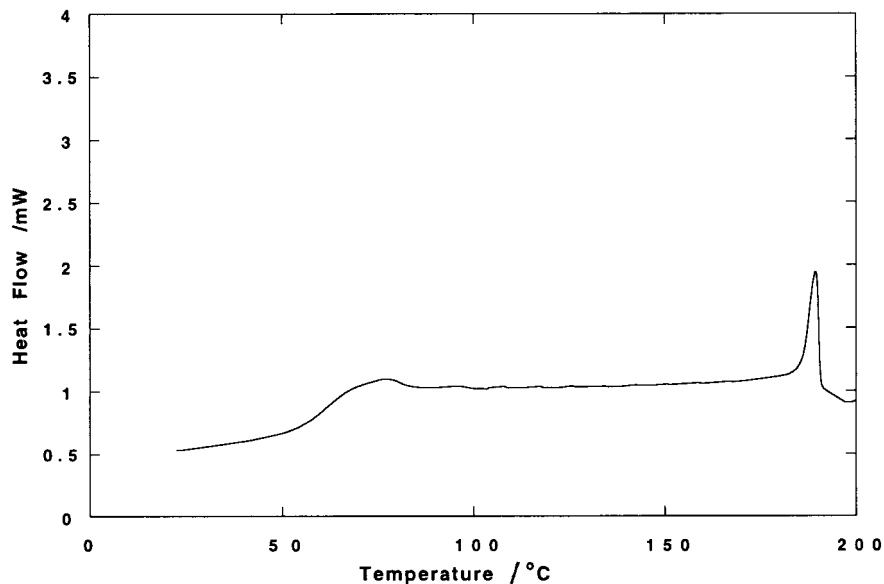
Figure 3.   Cooling DSC curve of compound 1 at 10°C min^{-1} after clearing. Increasing numbers refer to exothermic heat flow.

sapphire substrates as shown in figure 4. The IR spectra from this class of compounds is relatively free from vibrations from the –CH$_3$ and –CH$_2$– stretches at 2800–3000 cm^{-1} (3.6–3.4 µm) to the strong ester stretch at approximately 1750 cm^{-1} (5.7 µm). Between these features, a strong dip in the transmission is observed at 2400–2500 cm^{-1} for compound 1, corresponding to a wavelength of 4.0 µm. This band is attributed to reflection of radiation at this wavelength. Compounds with much stronger helical twisting power, including compound 2, do not exhibit this feature at this wavelength as indicated in figure 4. A shift in the notch wavelength minimum with concentration for cholesterol

also verified that this feature is due to the inherent cholesteric nature of the material and not to particular intrinsic molecular absorptions. Little difference between films formed on either substrate is observed, except that at lower wavenumbers (< 1500 cm^{-1}) the sapphire films become non-transmissive due to substrate absorption. In general, the sapphire films exhibited a lower transmission in the 4000 cm^{-1} region, which is attributed to increased loss due to Fresnel reflections [16]. In particular, the refractive index difference between sapphire (1.77) and compound 1 (1.55) is much larger than that between CaF$_2$ (1.44) and compound 1. The notch depth for these films is approximately 0.20–0.22 optical density (OD)
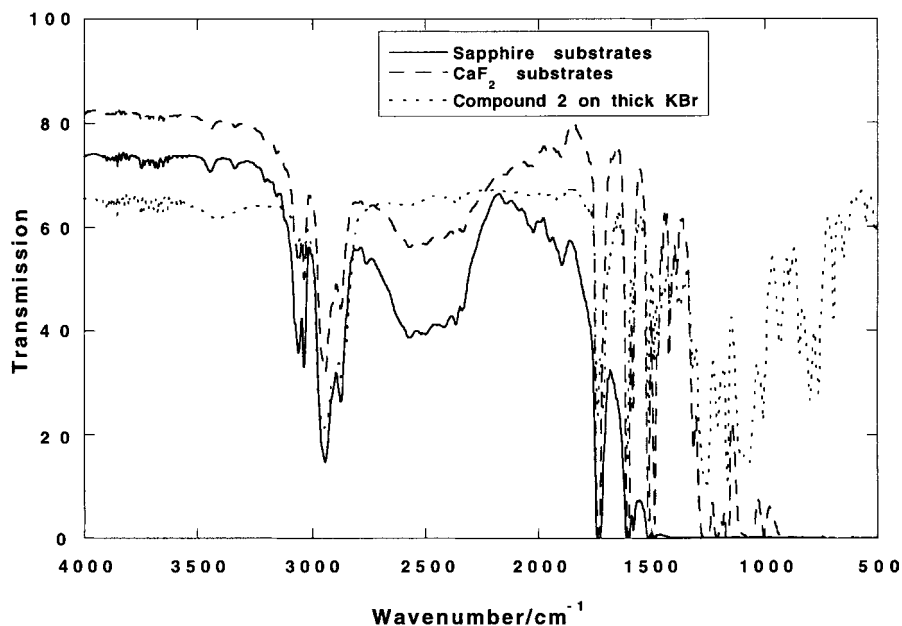

Figure 4.   IR spectra of processed films of compound 1 on CaF$_2$ and sapphire substrates, revealing a decrease in transmittance due to selective reflection at approximately 4.0 mm. The IR curve of compound 2 sheared on KBr plates without controlled thickness is shown, to confirm the notch is due to selective reflection and not to inherent IR absorption from this class of compounds.

which is approximately 2/3 of the expected theoretical value. A maximum decrease in transmission of 50% due to selective reflection would yield a notch with a 0.30 OD depth. These notch depth values were determined using the peak height and the 'baseline' value at $4000 \, cm^{-1}$. The bandwidth of the selective reflection peak was approximately 700–750 nm FWHM. Using a refractive index of 1.55, the pitch thus corresponds to 2580 nm and the birefringence, Δn, is calculated using equation (2) to be approximately 0.25:

$$\Delta \lambda = \Delta n P. \qquad (2)$$

To the eye, the best films look clear with only minor defects visible. Under the microscope, the same films exhibit oily streaks characteristic of cholesteric mesophases frozen into a glassy state [17].

The ability of these films to differentiate polarization states was measured using the simple device shown schematically in figure 2. Films formed from cholesterol-based mesogens are expected to reflect left-handed radiation and transmit right-handed radiation [18]. The variable waveplate used in the experiments allows one to convert the incoming linear polarized light into right- or left-handed polarization, and measurements are performed using both handednesses separately. The measured contrast ratios varied from 3:1 to 70:1 depending on the uniformity, or conversely the defect content of the film. The largest values were obtained for single substrate films which we partially attribute to decreased Fresnel losses from air/substrate and substrate/compound reflections. However, the ability to achieve large contrast ratios consistently was poorer for single substrate films. Standard reflection from air/substrate interfaces causes a 180° phase change in the polarization state of incoming radiation, thereby reducing the effectiveness of single polarization reflection [16]. Since the double substrate films have twice as many interfaces, the increased Fresnel reflections cause a decrease in the measured contrast values. Double substrate films gave more reproducible notches, but overall the contrast ratios were lower with the largest being 35:1. It is important to note that these contrast numbers are not corrected for Fresnel reflections [16], expected to contribute significantly to a decrease in contrast ratio, and are therefore lower estimates of performance. This is especially true for the double substrate films considering the large index difference between the substrates and this class of materials. The large measured (uncorrected) values of the contrast ratio for these glassy siloxane compounds suggest that very high contrast polarization could be obtained through the use of suitable substrates and/or the use of antireflective coatings. A contrast ratio of 70:1 compares very well with the quoted values of 42:1 for two commercial wire grid polarizers [19].

Compared with compound 2, uniform films of the large pitch compound 1 were considerably more difficult to achieve consistently. Much higher defect densities were consistently observed in films of compound 1 than typically observed for compound 2. Films were best produced by shearing at 150°C followed by slow cooling $(dT/dt = 2°C \, min^{-1})$ to ambient temperature by turning off the hot plate. Annealing of the resulting films for 24 h at 100°C yielded the most macroscopically homogeneous films. A trade-off between thickness and clarity was observed as the thinnest films looked the best visually, but also provided somewhat poorer values of contrast ratio than thicker films. The somewhat weaker reflectivity is expected as only 5–6 pitch lengths are present in the 15 μm films, from which, according to theory, one would expect a slight fall in efficiency. Good films could be obtained on both CaF_2 and sapphire plates and the homogeneity (to the eye) was the best on double substrate films. Single substrate films occasionally yielded very nice reflection bands compared with double substrate films; however they were difficult to produce repeatedly.

During the initial examination of compound 1 to explore the reasons for these inconsistencies, a fingerprint texture was observed under the microscope upon cooling from the isotropic phase. Although observed in other cholesteric compounds, this texture has not been reported for this class of macromolecules. We can only speculate that this compound, due to free energy considerations, prefers to lie with its helix axis parallel to the substrates. For similar compounds with higher cholesterol content and thus stronger helical twisting powers, this behaviour has not been observed; rather, a very strong tendency to align in a low defect-density Grandjean texture (helix axis perpendicular to substrates) is observed. The microstructural tendencies of compounds 1 and 2 are shown in the optical micrographs of figure 5. Compound 2 exhibits a very tight focal conic texture upon cooling from the isotropic to cholesteric phase, as shown in figure 5(a). This is typical for this class of molecules with reflection bands in the visible and NIR. When sheared, this focal conic texture is easily transformed into a brightly coloured Grandjean texture with characteristic oily streaks running through the field of view. This behaviour is observed on the edge of figure 5(a) where flow has occurred. A very small displacement of one substrate relative to another spontaneously eliminates the focal-conic texture into large uniform Grandjean regions with a uniform colour as indicated in figure 5(b). Running through the field of view are the characteristic oily streaks whose density depends on shear rate and temperature.
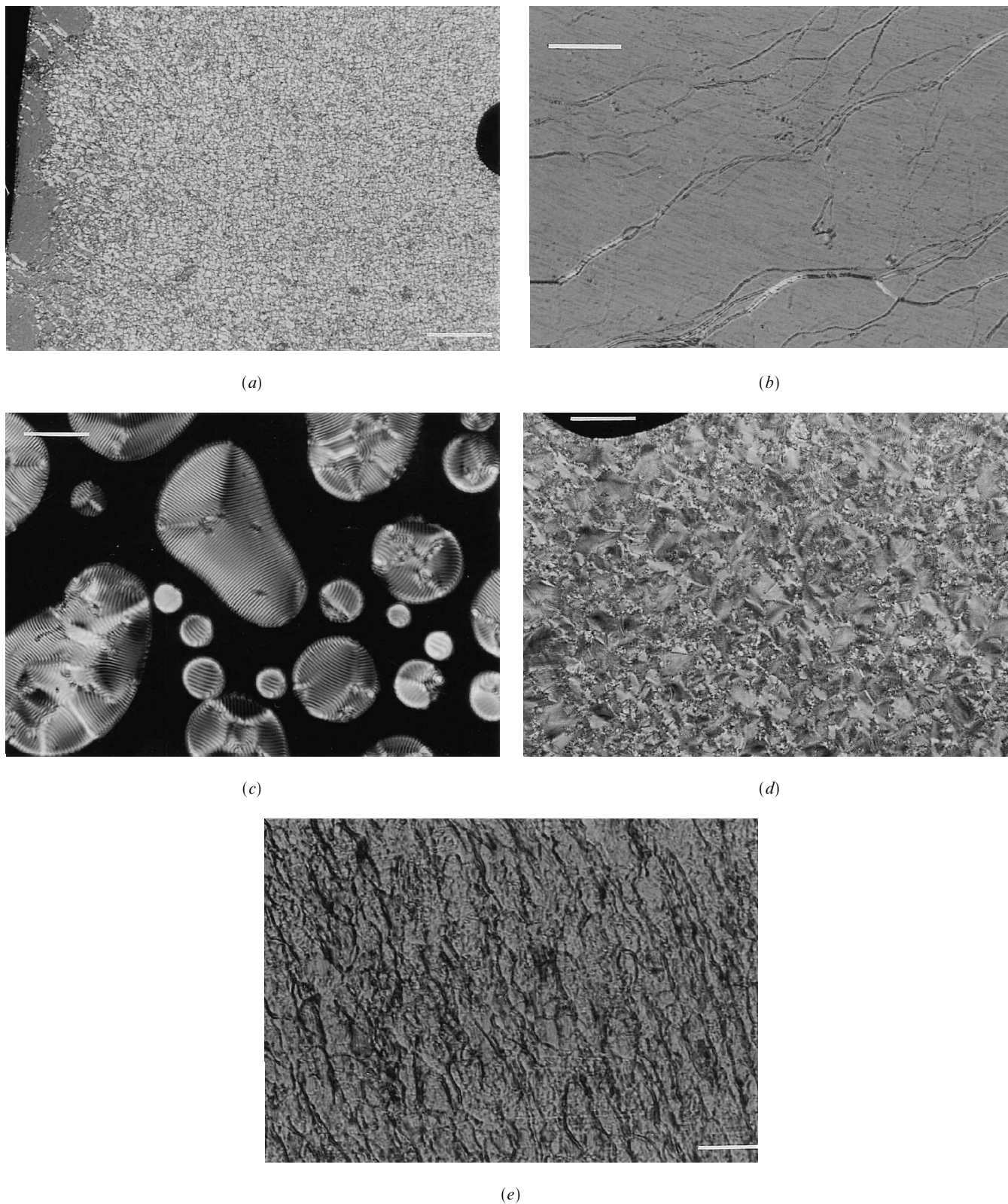
(*a*)

(*b*)

(*c*)

(*d*)

(*e*)

Figure 5. Polarized optical micrographs of compound 2 upon cooling from the isotropic phase (*a*) and after very gentle shear at 150°C (*b*). Compound 1 exhibits a fingerprint texture upon cooling from the isotropic phase (*c*) which persists upon cooling to 150°C (*d*). Upon shearing of compound 1 within the mesophase, a texture rich in disclinations is formed (*e*). White scale bars correspond to 80 µm except figure 5(*c*) where it corresponds to 40 µm.

However, upon cooling compound 1 from the isotropic state, a texture characteristic of a fingerprint texture is observed as shown in figures 5(c) and 5(d). Initially, parallel lines appear over the small individual LC domains which is suggestive of the helical axis lying in the plane of viewing. Further cooling results in a texture which is rich in fingerprint lines. These lines correspond to alignment where the helix axis is lying within planes parallel to the substrate planes, an orientation orthogonal to that of the Grandjean texture spontaneously observed in compound 2. Most importantly, however, is that upon shear from this orientation, a texture rich in defects is observed, as shown in figure 5(e). Large monodomain Grandjean textures observed in compound 2 were not obtainable from films of compound 1 wherein an initial fingerprint texture was present.

In the design of MWIR polarizers, one needs the helix axes to be perpendicular to the substrate and a low

defect content. Since the fingerprint texture is detrimental to facilitating this molecular orientation, the use of an alignment layer to control the surface anchoring characteristics of the compounds better was investigated. It was speculated that the poor consistency in obtaining good quality films was due in part to the poor packing induced from the fingerprint texture. This texture arises from a fundamental difference between the tendencies of molecules of compounds 1 and 2 to interact with glass surfaces as shown in figure 6. Both glass and CaF_2 substrates were coated with a polyimide alignment layers which were subsequently buffed. Since buffing typically induces homogeneous (planar) alignment in nematics, it was hypothesized that this would stabilize the proper orientation of the surface molecules at the substrates,
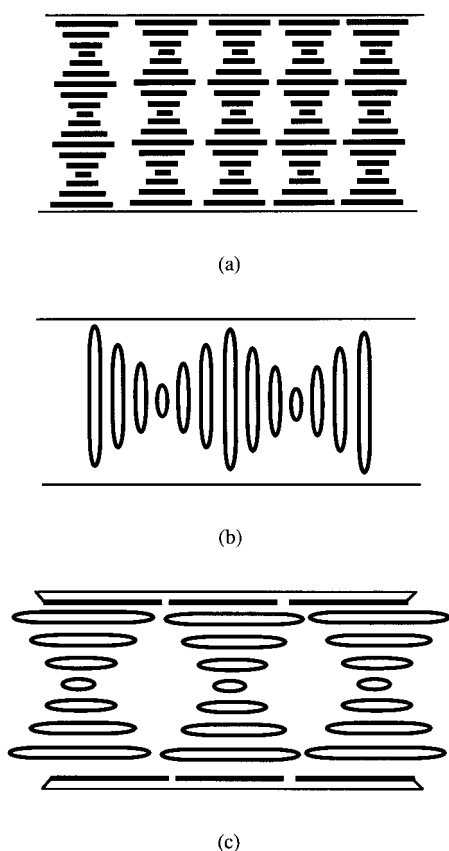

(a)

(b)

(c)

Figure 6. Schematic diagram of Grandjean alignment (a) of compound 2 (shorter pitch) which spontaneously occurs [see figure 5(b)], a schematic of the alignment (b) present in the fingerprint texture of compound 1 which occurs without surface treatment [see figure 5(c)], and the induced Grandjean texture of compound 1 obtained after surface treatment and buffing of the substrates (c). The dark horizontal lines represent a preferred orientation induced by the uniaxial buffing.
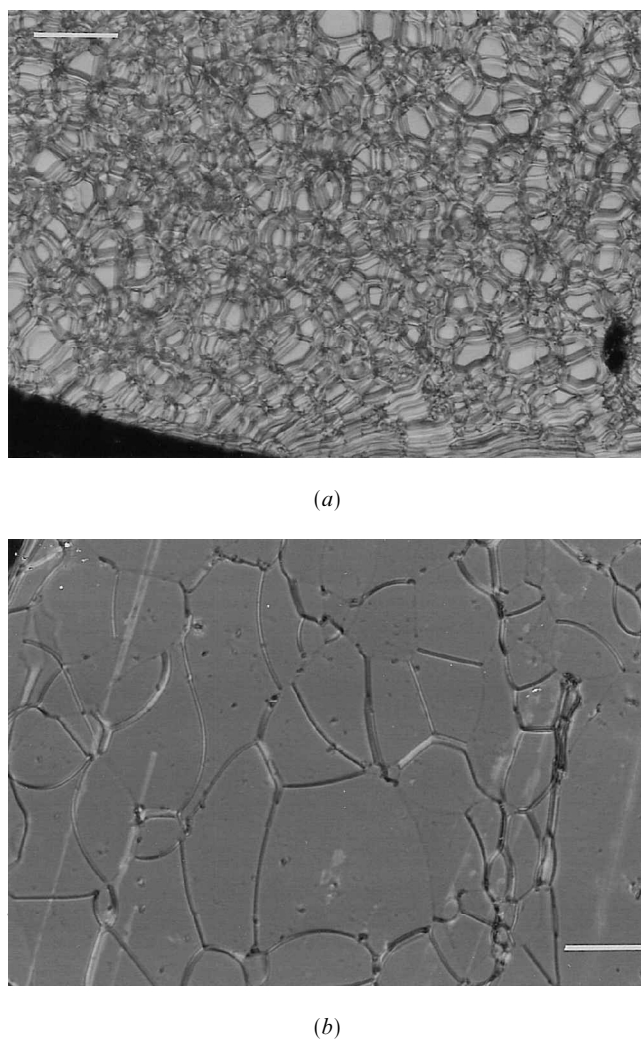

(a)

(b)

Figure 7. Polarized optical micrographs of compound 1 upon cooling from the isotropic phase on buffed substrates (a) and the alignment generated upon shear (b) on the buffed substrates. White scale bars correspond to 80 μm.

as indicated in figure 6. Starting from an initially homogeneous orientation, the mesophase would then propagate the helical twist across the cell gap in an orientation conductive to forming a Grandjean texture.

Figure 7 (*a*) indicates that films of compound 1 formed on the buffed glass slides do not exhibit the fingerprint texture. This texture was stable throughout the cholesteric mesophase range as single, isolated domains with no indication of fingerprint lines within domains

being observed. Upon shearing and annealing at 150°C for 24 h, films with large monodomain Grandjean textures were formed, as shown in figure 7 (*b*). Oily streaks were still present but in a greatly reduced density as compared with the non-buffed films shown in figure 5 (*e*). The buffed films consistently demonstrated uniform textures indicative of macroscopic alignment, while uncoated films yielded only small regions of uniform alignment. There was no discernible effect on compound 2 as well-aligned

(*a*)
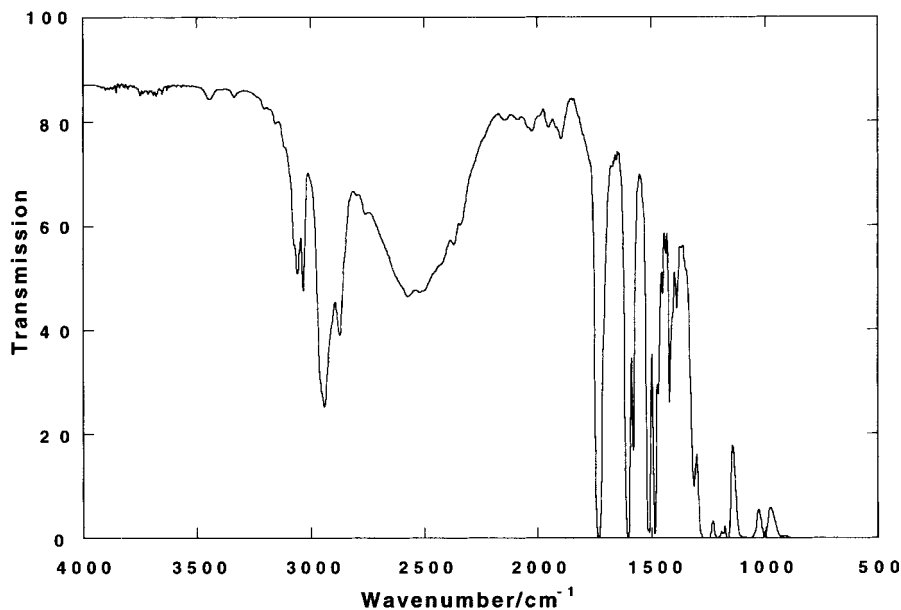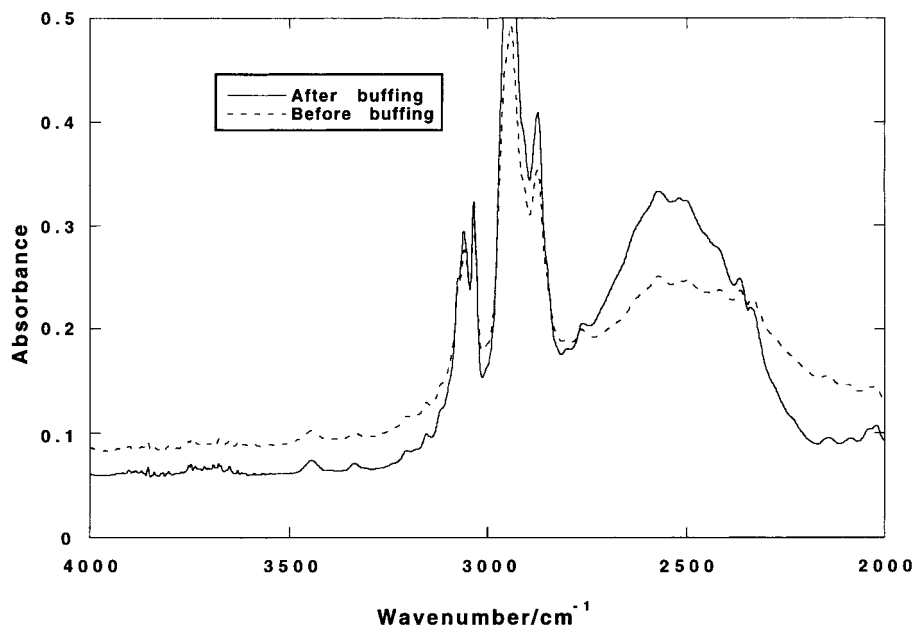
Figure 8. The IR spectra of compound 1 on buffed CaF2 substrates (*a*) in transmission and a comparison of the before and after buffing spectra (*b*) in absorbance units.

(*b*)

textures similar to figure 5(*b*) were observed for both buffed and non-buffed slides. Thus, it appears that the introduction of a simple alignment layer overcomes any tendency of the larger pitch cholesteric to adapt an unfavourable macroscopic orientation of the helical axes.

The MWIR selective reflection and polarization properties of films of compound 1 formed on buffed CaF_2 substrates were examined. The IR spectrum is shown in figure 8(*a*); the shape of the curve is similar to those shown in figure 4 but there are subtle differences as indicated in figure 8(*b*). The 'baseline' transmission is higher (lower baseline absorbance) and the depth of the notch deeper than those formed on unbuffed slides. This results in a notch depth of 0.28 OD which is very close to the maximum of 0.30 OD. The bandwidth of the notch is also slightly narrower at 600 nm. We attribute these traits to better macroscopic orientation of the cholesteric helix which results in less scattering and more effective reflection. The contrast ratio of the films formed on buffed layers was comparable to those measured previously. However, the use of buffed slides allowed for much greater consistency in maintaining the macroscopic orientation induced in the films.

4. Conclusions

The use of glass-forming liquid crystals which possess useful processing windows has been demonstrated to be effective in the formation of MWIR circular polarizers. The inherent ability of cholesteric liquid crystals to polarize transmitted light selectively has been coupled with the ability of excluded-volume core-based macro-molecules to form optical quality thin films wherein the induced macroscopic ordering is frozen. MWIR polarizers were formed with comparable size and device per-formance to commercially available wire-grid polarizers. The use of buffing layers was shown to stabilize the proper macroscopic helix direction for optimum polarizer performance.

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