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Lasing from chiral photonic band gap materials based on cholesteric glasses

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Glass-forming cholesteric liquid crystals were studied as promising dye-doped lasing materials at different pumping energies and temperatures. Cholesteric samples doped with laser dye pyrromethane 597 were pumped by Nd:YAG laser. Lasing was found to depend strongly on the vitrification rate of cholesteric samples, their temperature and multidomain structure. The lasing threshold and intensity as a function of thickness of cholesteric resonator are determined by two competing factors: narrowing of the band edge modes and increasing disorder.

1. Introduction

Coherent multiple scattering from the periodic helical structure in cholesteric liquid crystals (CLCs) produces a selective reflection band for light with the same sense of circular polarization as the helicity of structural anisotropy [1, 2]. Transmission of light of the opposite circular polarization is unimpeded by the chiral structure. Within the reflection band, light with the polarization that is reflected is evanescent and the density of states vanishes. Since emission is proportional to the density of states, emission from molecules doped into the CLC within the stop band is inhibited [3]. The modes that are suppressed within the band gap pile up at the band edge in a series of closely spaced narrow modes. The residence time for light within the CLC, and hence the opportunity for stimulated emission, is greatly enhanced at these band edge modes. This leads to low threshold lasing in narrow transmission resonances of dye-doped CLCs [3, 4] rather than in the broad reflection band, where lasing was initially expected [5–7]. It was proposed nearly 30 years ago that dye-doped CLCs would be an ideal lasing medium because light emitted near the centre of the reflection band would be strongly reflected within the structure resulting in lengthened resident times inside the medium [5]. Initial measurements of lasing far from the centre of the reflection band where reflection is at a peak were consequently interpreted as arising from inhomogeneities within the CLC [6, 7]. It was realized subsequently that lasing could not occur within the stop band because emission is suppressed. Instead lasing would occur in long-lived modes at the band edge [3, 4].

Lasing at the band edge is readily achieved in highly periodic single domain samples. Monodomain cholesteric samples can be created by filling planar cells with liquid chiral nematic. For many purposes it is advantageous to use more stable solid samples whose structural integrity is insensitive to temperature changes and mechanical vibrations. Recent studies of laser dye dopants in various polymer matrices revealed that they could emit light more efficiently than in liquids [8, 9].

Solid cholesteric materials might be created either (1) by crosslinking acrylate or methacrylate monomers, among others, in a particular cholesteric material [10–12], (2) by synthesizing side or main chain polymers which may form a glassy state with frozen cholesteric structure [13–15], or (3) by synthesizing cyclic glass-forming oligomers [16–19]. Glass-forming cholesteric liquid crystals can be readily processed into ordered solid films. Among the chiral glass-forming liquid crystals that have been synthesized [16–19] are chiral cyclic silicones synthesized by Kreuzer [18–19]. These are promising materials for a variety of optical applications including lasing. Recent studies have

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examined lasing in doped [4, 6, 7] and neat [20] thermotropic liquid crystals, doped liquid crystalline elastomers [21] and doped lyotropic polymers [22]. Here we report, for the first time, lasing in vitrified cholesteric liquid crystals based on cyclic silicones produced by Wacker-Chemie. These cyclic cholesteric silicones are of interest because they easily form cholesteric glass with a selective reflection band lying in the visible region of the spectrum. The goal was to study lasing in cholesteric glasses and to find a relationship between lasing characteristics and liquid crystalline properties of cholesteric glass.

The position of the selective reflection band, as well as the texture of cholesteric samples, can be affected by a number of variables, among which temperature and rate of transformation into the solid state are the most important. Because of the stability of these polymeric materials, lasing can be studied over a broader range of temperature than in thermotropic liquid crystals. We investigate the effect of various material parameters upon the lasing properties of cholesteric materials. We also examine the influence on lasing of various defects of vitrified cholesteric liquid crystals. We find that the multi-domain structure of cholesteric materials has a significant influence upon the optical properties of cholesteric liquid crystals. It is therefore difficult to draw conclusions regarding lasing in solid cholesteric materials without careful consideration of structural imperfections. An analysis of the shape of the selective reflection band, optical microscopy observations, as well as lasing measurements provide a qualitative understanding of the role of defects.

This paper is organized as follows: § 2 describes the preparation procedure and experimental set-up. The characterization of the selective reflection band and morphology of cholesteric samples are discussed in § 3, followed by a description of lasing from the cholesteric samples in § 4; conclusions are presented in § 5.

2. Experimental

All compounds were obtained from Wacker-Chemie and used as received. The synthesis and characterization of cyclic oligosiloxanes has been described elsewhere [18, 19], their structure is shown in figure 1. The cholesteric films, cast from a solution in toluene on a glass substrate covered by a rubbed polyimide layer, were doped with a laser dye with an emission band in the wavelength region of the reflection band of the cholesteric oligosiloxane. The laser dye was pyrromethane 597 supplied by Excition. Pyrromethane 597 is highly miscible with cholesteric material, but only a low concentration of dye was used in order to avoid aggregation of dye molecules, and to achieve an appropriately low absorption of pump radiation so that excitation would be distributed throughout the sample. The absorption and emission peaks of the dye in toluene solutions were at 526 and 575 nm, respectively.

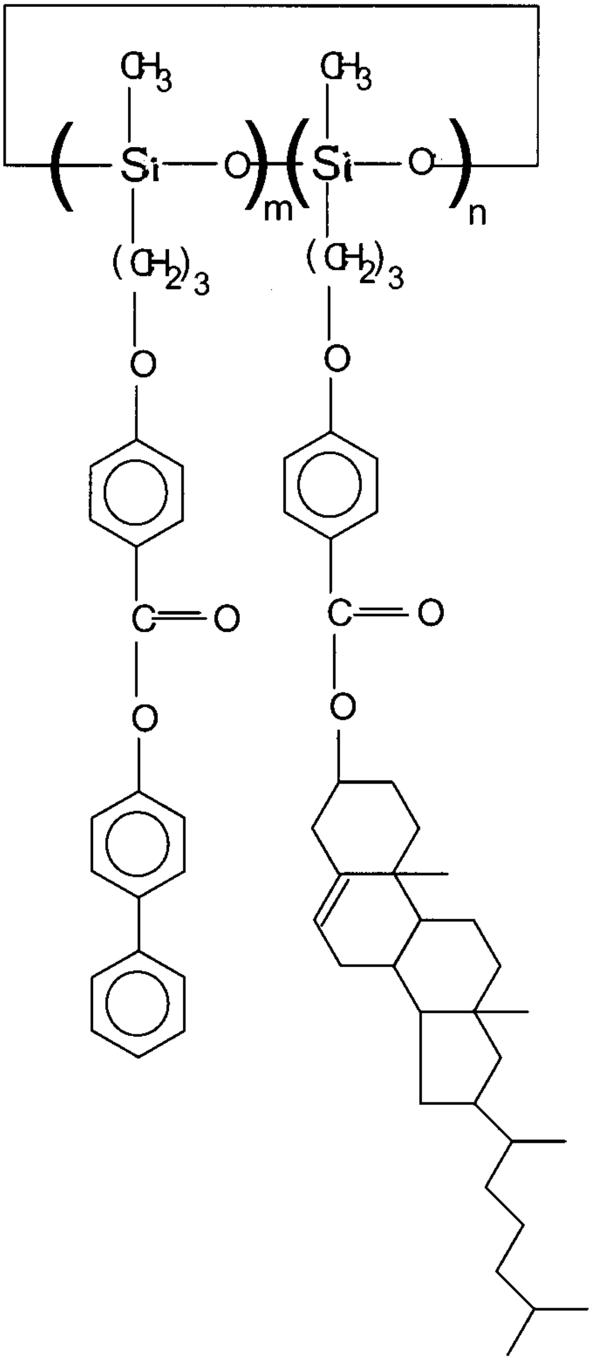


Figure 1. Chemical structure of cholesteric liquid crystal.

After evaporation of the solvent, the cast films were covered with a second glass slide at 120°C and the cholesteric cells were sealed. Sample thickness ranged from 8 to 150 μm and was controlled by two plastic strip spacers and/or glass beads. All cells were annealed at 130°C for two hours before being cooled to room temperature. Two cooling methods were used. Slow cooling was performed by reducing the temperature of the hot stage to room temperature over a period of 2–3 h (S-samples). Fast cooling was performed by placing samples heated to 130°C in a water bath at room temperature (F-samples). Slow and fast cooled samples displayed quite different structural and lasing properties.

A Nd:YAG laser (Photonics Industries, Inc.) was used to excite the dye near its absorption maximum. The emission was focused on the entrance slit of a monochromator. The pump pulse was approximately 45 ns in duration; the pump beam was focused to a spot with a diameter of approximately 140 μm. A 20 cm focal length lens was used to collect the light and to focus it on the entrance slit of the monochromator. Spectrally resolved emission was recorded with a CCD detector attached to the monochromator (CVI Digikrom).

3. Selective reflection and morphology

At room temperature, the selective reflection band of S-samples was centered near 545 nm. Fine structure at the edges of the selective reflection band such as in monodomain CLCs [23] was observed only in samples as thin as 8–15 μm (figure 2). In thicker samples, fine structure could not be observed at the band edges. The centre of the selective reflection band did not shift with temperature (figure 3). The multidomain structure of the planar cholesteric material washed out the sharp selective reflection bands as well as oscillations near the band edge observed in planar monodomain samples [23]. A slight non-uniformity in the pitch across the sample also tended to wash out the fine band edge structure. This suggests that samples thicker than 15–20 μm have a multidomain or so-called imperfect planar structure. At temperatures above the glass transition temperature of $T_g = 57°C$, the selective reflection band narrows and sharpens with increasing temperature (figure 3). The same behaviour is observed for all samples prepared by slow cooling, independent of thickness. A plot of the temperature dependence of the width of the selective reflection band is shown in figure 4.

A cholesteric medium may be regarded as consisting of a large number of thin birefringent planar sections with ordinary and extraordinary refractive indices n_o and n_e in the plane, respectively. The wavelength of the centre of the selective reflection band is given by:

$$\lambda_c = P\left(\frac{n_o + n_e}{2}\right) \qquad (1)$$

where P is the pitch of a cholesteric helix. Since λ_c is found not to vary with temperature (figure 3), either both the sum $n = n_o + n_e$ as well as the pitch P are independent of temperature, or changes in these qualities compensate. The width of the selective reflection band $\Delta\lambda = P(n_e - n_o)$ is proportional to the

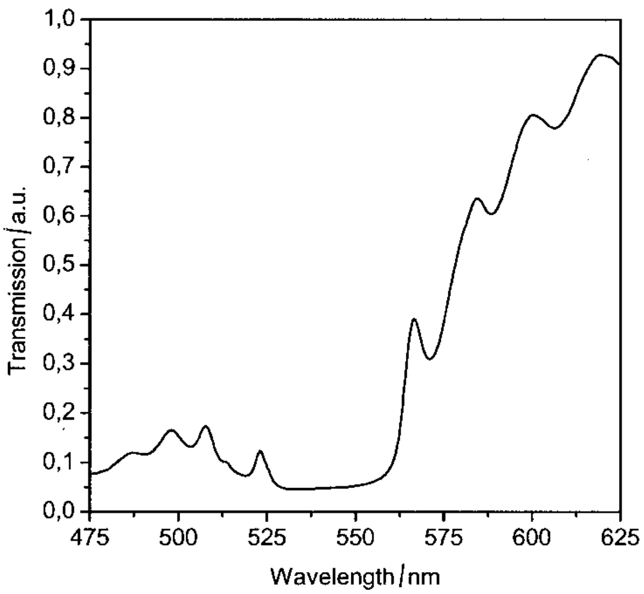


Figure 2. Selective reflection band of very thin S-film (about 8 μm) displaying a fine edge structure.

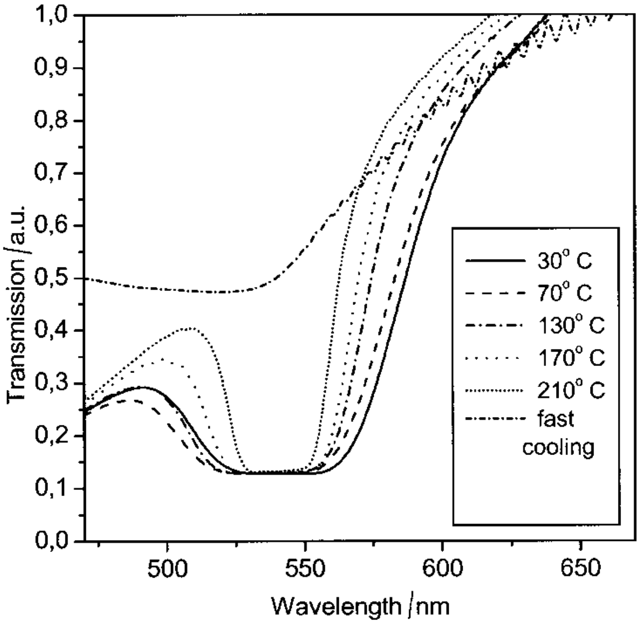


Figure 3. Selective reflection band of thin S-film (30 μm) at different temperatures.

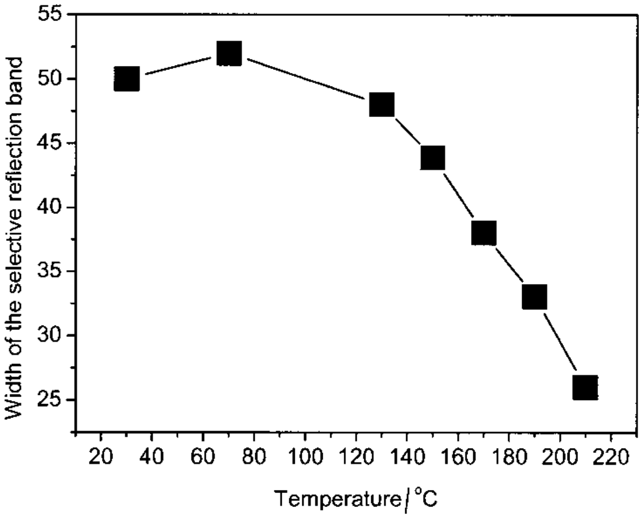


Figure 4. Narrowing of the selective reflection band as a function of temperature.

birefringence

$$\Delta n = n_e - n_o. \quad (2)$$

The birefringence of nematic layers as a function of temperature is plotted in figure 5. Finkelmann *et al.* have shown that the order parameter *S* of each nematic layer in a CLC is given by [24]:

$$S = C^* \left[\frac{n_e^2 - n_o^2}{n^2 - 1} \right] \quad (3)$$

where *C* is a constant depending on the molecular polarizabilities of mesogenic molecules. For Wacker cholesteric silicones, the measured width and central wavelength of the selective reflection band yield $n_o = 1.520$ and $n_e = 1.665$. The same data were obtained by measuring the refractive indexes of the cholesteric material. Due to the similarities in chemical structure of Wacker silicones and the polymers described by Finkelmann *et al.* [24] we assumed that changes in the averaged refractive index with temperature are the same for these materials, about $10^{-4} K^{-1}$. Thus the only adjustable parameter *C* determines the absolute value of order parameter *S* at room temperature. If we further assume that $S_o = 0.7$ (a typical order parameter value for a planar layer of nematic on rubbed polyimide), we obtain the temperature dependence of the order parameter *S* (figure 6). The temperature dependence of the order parameter of Wacker polymers (curve 1, figure 6) is similar to that of polymers described in [24] (curve 2, figure 6).

In F-samples, the selective reflection band is shifted towards shorter wavelengths and becomes much broader than in S-samples and exhibits smooth edges (figure 3). Visual examination of the sample reveals areas of different colour. Thus, fast cooling results in inhomogeneous samples. The heating of rapidly cooled samples restores the original selective reflection properties.

The morphologies of thin and thick films were found to be quite different. Optical microscopy revealed defects in all the cholesteric samples in this study,

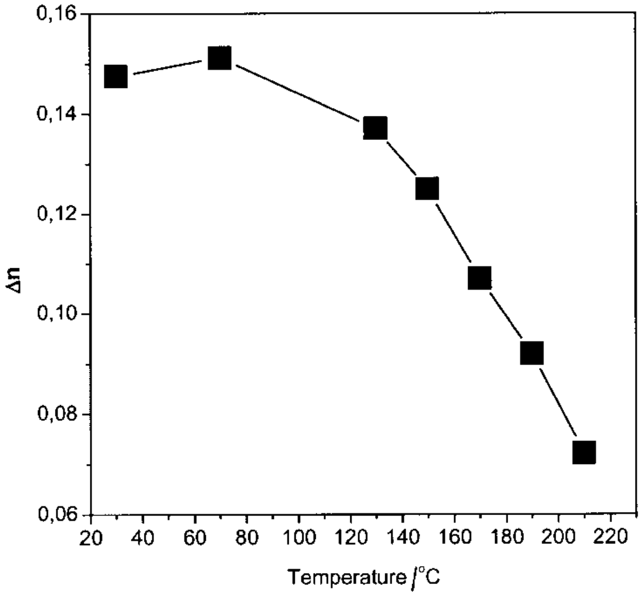


Figure 5. Birefringence of the nematic layer as a function of temperature.

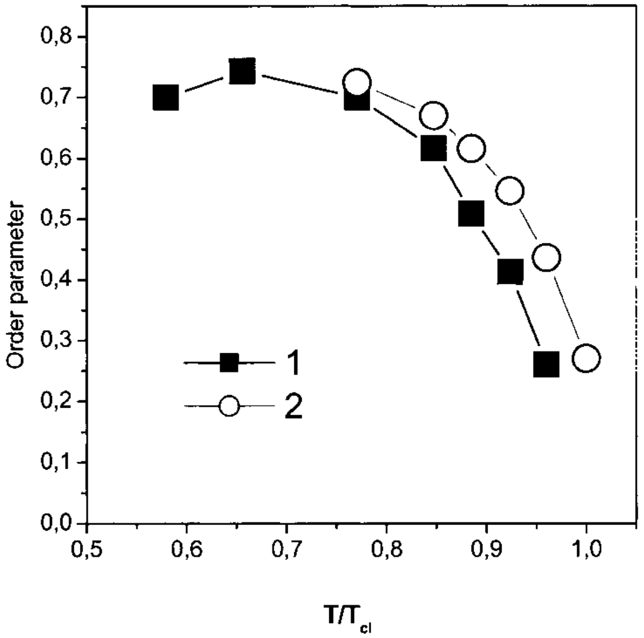
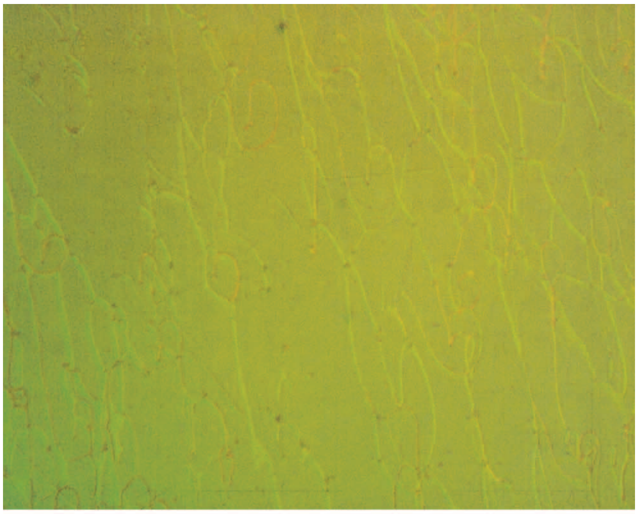


Figure 6. Order parameter of the nematic layers at different temperatures: 1—calculated data for S; 2—experimental data from [23].

figures 7 (*a*, *b*). The pictures were taken in reflection mode to make the analysis of morphology easier. The size of the domains in thin samples is larger (typically, 100 μm) and the distribution of the domains in thin samples seems to be much more uniform than in thick samples. Disclinations, oil-streak defects and non-uniformities in the pitch and helix orientation with respect to the film plane are the prevailing type of defects in thin films, figure 7 (*a*). The domains are easily recognized by the distinctive colours of regions with different orientation of the helical axis. In some samples, the colour changes gradually, indicating that the direction of the helix and/or pitch also changes gradually within the sample. In thick samples, the defects tend to form clusters; the areal density of defects is much greater, figure 7 (*b*), and the domain size is smaller, typically 10 μm.



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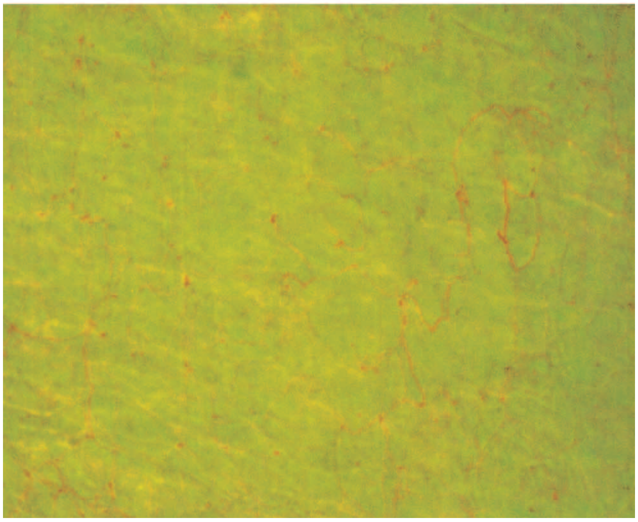


(*b*)

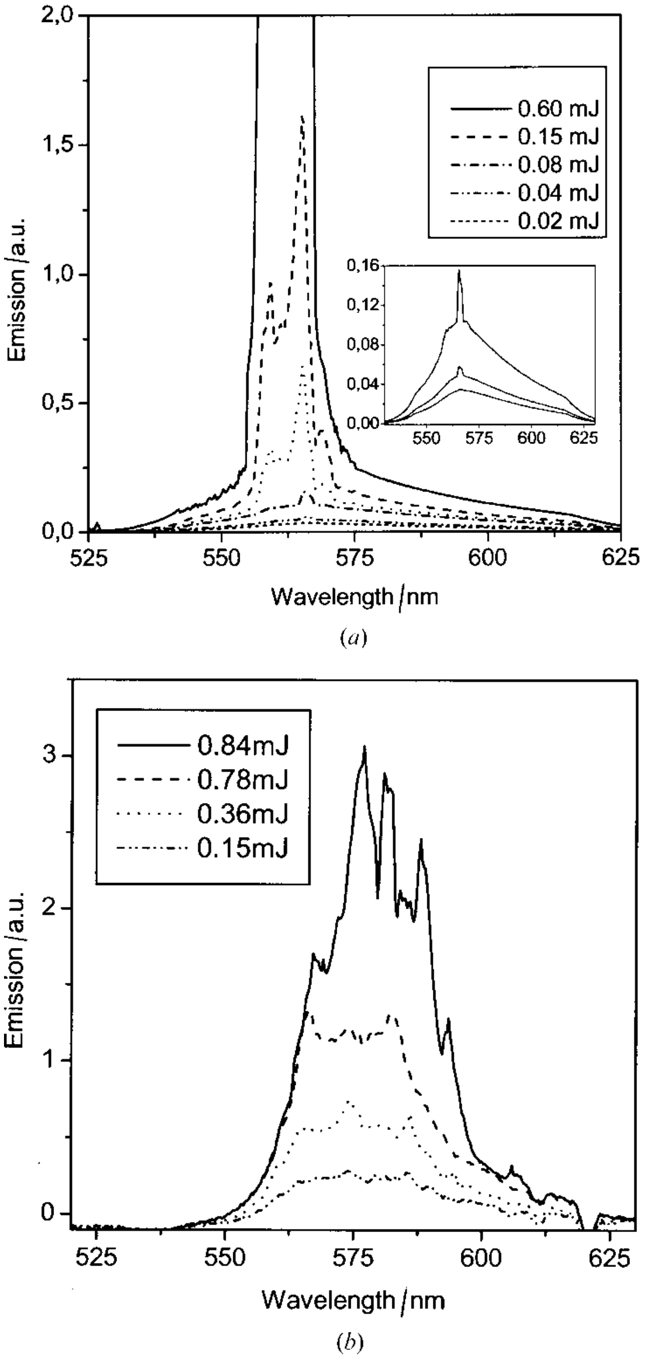
Figure 7. Texture of (*a*) thin and (*b*) thick S-films.

Upon heating, the colour of different domains within S-samples becomes more uniform. At the same time, the edges of the selective reflection band become sharper. This indicates that heating improves the planar orientation of the sample.

4. Lasing

The lasing properties of S- and F-films were found to be very different; the latter display much higher laser threshold. S-films with thickness under 10 μm displayed narrowing of spontaneous emission inside the broad first mode but did not lase. The width of the mode is inversely proportional to the photon lifetime inside a cholesteric resonator. Thin samples with a wide first mode are poor resonators. However, the width of the first mode decreased with increasing sample thickness, indicating that the photon dwell time inside the sample increased. Lasing was observed from samples with a thickness of about 15 μm at relatively high pumping energies of about 0.3 mJ. In thicker samples (25 μm), lasing was achieved with pumping energies below 0.1 mJ. However in much thicker samples, the lasing threshold increased again. In all samples the concentration of dye was less than 0.15%. The absorption coefficient was estimated to be *c*. $7\times10^{3}\,\mathrm{mm}^{-1}$. In 35 μm thick sample about 50% of the pumping beam was absorbed at a dye concentration of 0.14%. From a theoretical standpoint the most favourable conditions for lasing are achieved at an absorption of 50% of pumping radiation; the increase of thickness of an ideal cholesteric resonator should result in a dramatic decrease of lasing threshold [3–7]. In our experiments the lowest laser threshold was achieved in 25–30 μm thick samples. For thicker samples, the disorder and imperfect planar structure of the samples result in a higher laser threshold.

Let us consider the lasing behaviour of thin and thick samples in more detail. Typical room temperature emission spectra obtained from a thin sample are shown at figure 8 (*a*). Lasing peaks are narrow and appear on the top of a broad emission band ranging from 535 to 625 nm. At low pumping energy a single lasing peak on the top of the broad emission band is seen on insets in figures 8 (*a*), and 9 (*a*). At higher pumping energy lasing occurs closer to the long wavelength edge of the selective reflection band in the range 12–16 nm (compare with the position of the selective reflection band in figures 2 and 3). Note that at high pumping energy each peak consists of several modes. The width of a single mode is close to 2–3 nm. The resolution of our monochromator was limited to

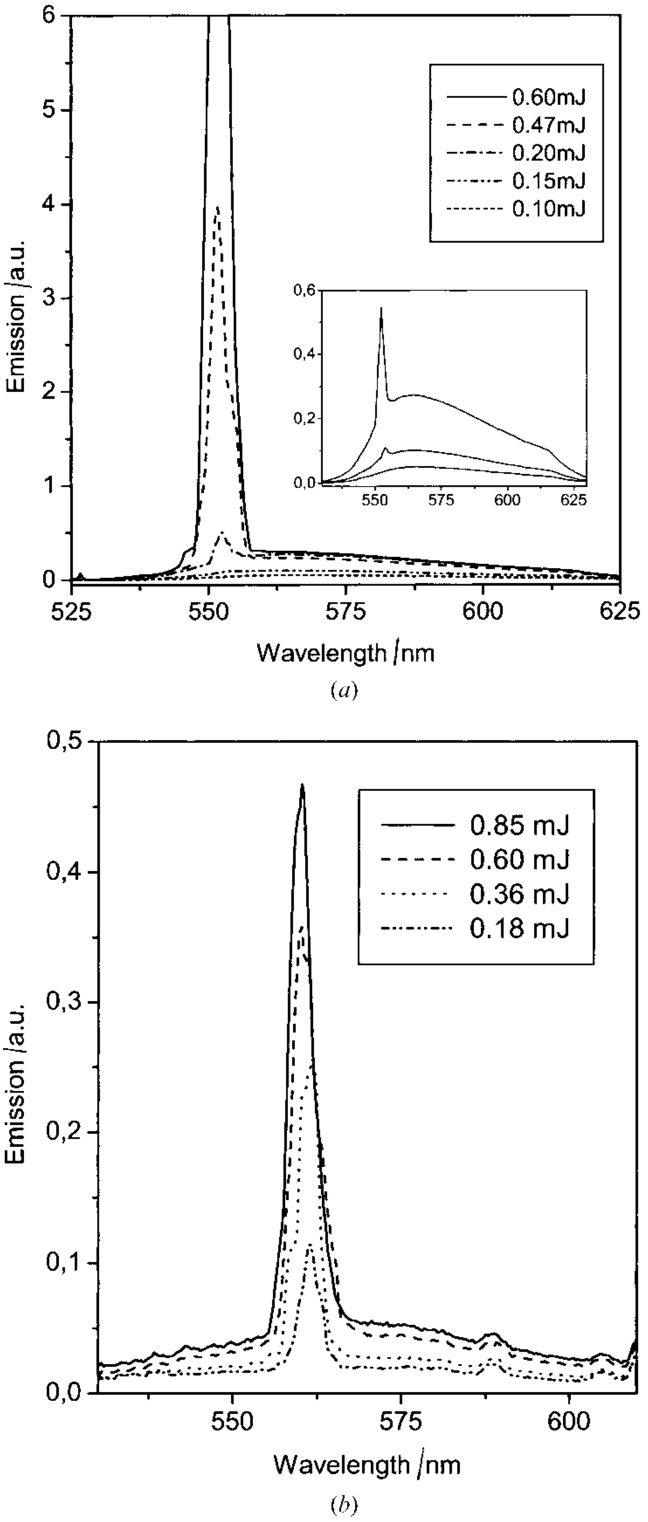


(a)

(b)

Figure 8. Lasing from (a) thin 30 μm, and (b) thick 130 μm slow cooled samples.

c. 0.6 nm; more closely spaced modes could not be resolved. At high pump power, several peaks are observed in the broad emission band, while at low pump power a single peak is observed inside the selective reflection band close to the edge. In thick S-samples at room temperature, lasing occurs over a much broader range of wavelengths, typically about 35–40 nm, figure 8 (b). However, the width of lasing modes is again close to 2–3 nm. This is additional evidence that lasing originates in single domains with narrow band edge modes.



(a)

(b)

Figure 9. Lasing from (a) thin 30 μm and (b) thick 130 μm samples at 125°C.

The difference in lasing behaviour observed in thin and thick S-samples can be explained in terms of structural differences between these samples. The orientation of various domains is more planar in thin samples, so that the conditions for lasing more closely resemble those in monodomain samples in which lasing occurs in a single mode or in a small number of closely spaced modes at the band edge [3]. In addition, the number of defects and domains in a thick sample is higher, so that several domains may be pumped in both the transverse and longitudinal directions as the size of pumping beam (140 μm) becomes much larger than the size of a single domain. In thick samples the large number of domains is excited by the pumping beam. However, the smaller size of the domains in thick samples results in a higher lasing threshold and its confinement to a smaller space. This lowers lasing intensity and raises spontaneous emission, resulting in the appearance of less distinctive lasing peaks on top of the emission band. Light scattering between disoriented domains can also give rise to the formation of closed photon paths inside the material, resulting in random lasing [25, 26]. However we did not aim to study the possibility for random lasing in thick samples in detail.

Lasing behaviour does not change up to the glass transition temperature T_g. Significant changes are observed, however, at temperatures higher than $T_g+20°C$. The number of lasing peaks in the emission spectrum decreases and the lasing frequency range narrows in all samples, figure 9 (*a*, *b*).

At temperatures higher than T_g, the cholesteric liquid crystal becomes a viscous liquid and the sample texture changes as a result of material flow inside the LC cell. The changes observed in the polymer texture and transmission spectrum suggest that the orientation of domains becomes more planar and the number of defects decreases as the temperature increases. This results in fewer lasing peaks and in their appearance closer to the edge of the selective reflection band.

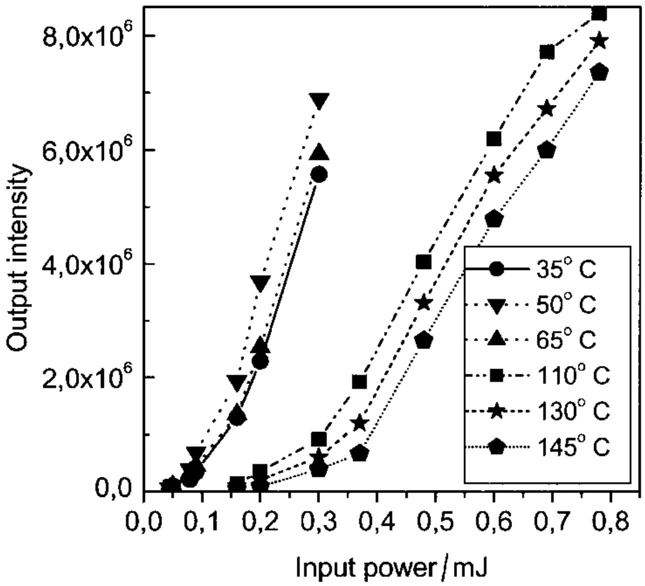


Figure 10. Lasing intensity as a function of input energy.

The lasing intensity as a function of pump energy is displayed for different temperatures in figure 10. The two sets of curves display different lasing behaviour. Curves of type I ($T<T_g+20°C$) corresponding to lasing from a glass have a relatively low lasing threshold. Type II curves ($T>T_g+20°C$) correspond to lasing from cholesteric liquid crystals heated above the glass transition temperature. One might expect that improved texture may also reduce the lasing threshold and improve the efficiency of lasing. However, the lasing threshold is higher in melted samples than in glassy samples. The transition from type I to type II occurs near 80°C; thus the lasing characteristics below and above $T_g+20°C$ are quite different. Interestingly, at this temperature there is a sharp drop in viscosity of the cholesteric liquid crystal (figure 11). Above 80°C the viscosity drops by three orders of magnitude, allowing the structure of the cholesteric liquid crystal to adjust itself to the planar boundary conditions imposed by the rubbed polyimide-coated glasses. The less viscous the cholesteric sample the more nearly planar the structure. This is also evident from the sharpening of the edges of the selective reflection band at elevated temperatures (figure 3). The greater the planarity of the structure and the lower the density of domains, the smaller the number of lasing peaks observed and the greater the

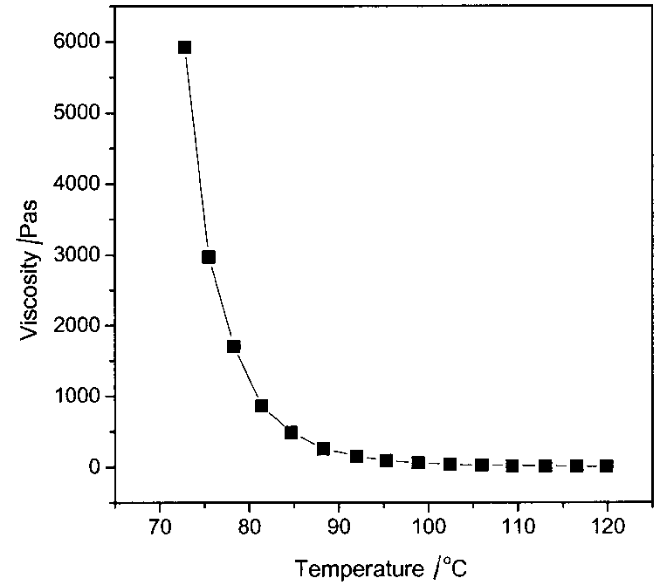


Figure 11. Viscosity of the sample as a function of temperature.

coherence area of the laser emission. The quenching of cholesteric samples at high temperature results in a reduction in the number of defects. For example, quenching of cholesteric samples for 24 h at 130°C decreases the number of defects by a factor of five. We did not aim to study this effect in detail. A recent study on cholesteric liquid crystals performed by Lubensky has revealed a slow coarsening of the oily-streak network in viscous samples [27]. It was argued that this rate is inversely proportional to the viscosity of the cholesteric.

A number of factors tend to raise the lasing threshold in cholesteric samples at elevated temperatures. The first is an increase in the rate of relaxation of excited dye molecules in a less viscous liquid matrix when it transforms from solid glassy state to liquid crystalline state at elevated temperatures. The cause of this 'matrix effect' is the high mobility of chemical groups in the pyrromethane molecule and the defreezing of rotational degrees of freedom in the liquid state. The magnitude of the effect is high for some dye molecules [28]. For example, rhodamine B dissolved in solvents with varying viscosity (methanol and glycerol) displays a decrease in photoluminescence of about two orders of magnitude as the viscosity increases. The changes of pyrromethane photoluminescence properties in the cholesteric matrix were studied by measuring the photoluminescence of highly disordered cholesteric samples as a function of temperature. The laser beam was defocused and the excitation energy kept low to avoid lasing. The dependence of photoluminescence intensity on temperature is shown in figure 12. The most dramatic changes in photoluminescence of pyrromethane molecules occur in the temperature range 60–100°C. At higher temperatures the change is more gradual with increasing temperature. The range 60–100°C is characterized by a sharp decrease in sample viscosity (figure 11). This suggests that the transformation from the solid to the liquid state ('matrix effect') leads to a decrease in photoluminescence.

The second and most important factor that should raise the lasing threshold is the change of order parameter of the liquid crystal with temperature. The width of the band edge modes for a single domain has been calculated by simulations of light propagation in cholesteric liquid crystals with the same refractive indexes and pitch as Wacker cholesteric material. The model used in the calculations was described in [3, 4]. The width of the first mode increases as the order parameter S decreases. The decrease of the mode width results in a shorter photon lifetime and a reduction of the quality factor of the cholesteric resonator (so-called Q-factor) for the mode given by

$$Q \approx \lambda/\Delta\lambda \qquad (4)$$

where λ is the wavelength of the mode and $\Delta\lambda$ is its width. The calculated Q-factor for the first mode as a function of order parameter is plotted in figure 13. The

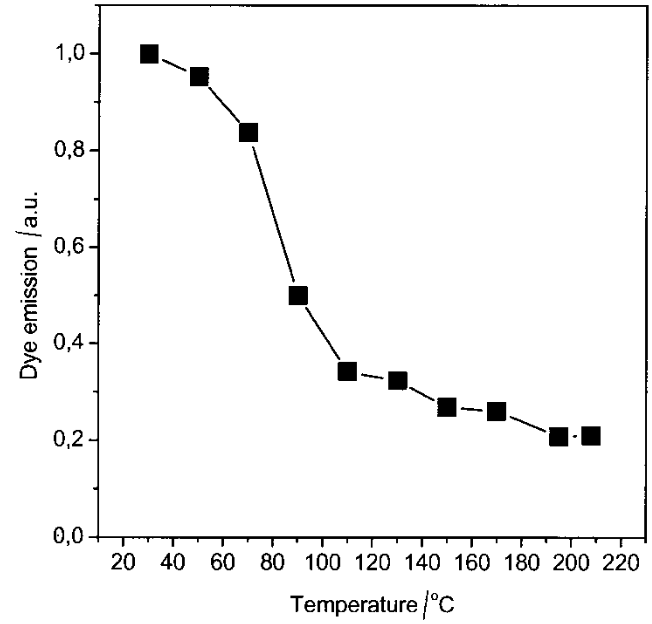


Figure 12. Photoluminescence intensity, at an excitation energy much lower than lasing threshold, as a function of temperature.

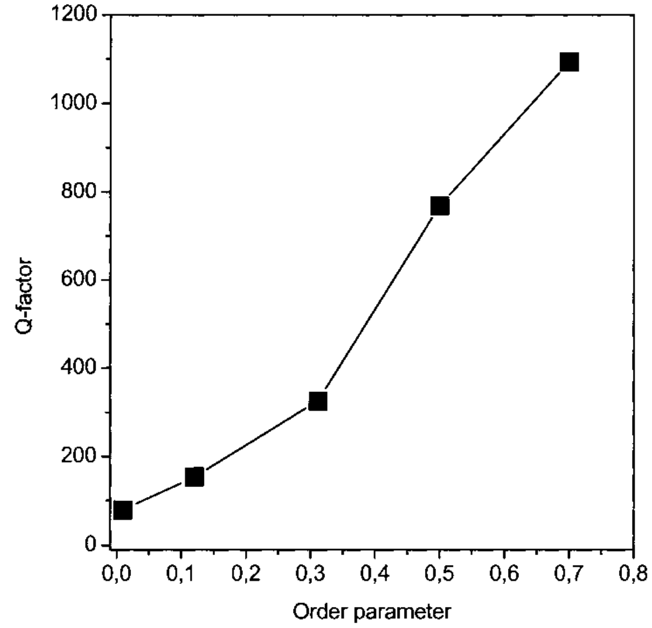


Figure 13. Simulated width of the first mode at different order parameters.

Q-factor is proportional to the photon dwell time, $\tau = Q/2\pi\nu_c$: the longer the photon dwell time, the lower the lasing threshold. Calculations show that the *Q*-factor falls by a factor of 2–3 when the order parameter decreases by 0.4 at elevated temperatures. The decrease of the *Q*-factor should result in an increase of lasing threshold roughly by the same factor. Thus, even though increased structural uniformity ordinarily suppresses the laser threshold, this trend is countered by reduced photoluminescence efficiency and a lowered photon dwell time in the medium at elevated temperatures.

The decrease of the mode width with temperature is a common feature of all thermotropic cholesteric samples undergoing a nematic–isotropic transition. This factor will therefore raise the lasing threshold in all thermotropic polymer and low molar mass liquid crystals. Lyotropic liquid crystals represent another class of materials, which do not necessarily have the same temperature behaviour. For example, in the case of lyotropic liquid crystals formed from ionic surfactants, liquid crystal properties are only weakly dependent on temperature [29]. Raising the temperature may leave the width of the modes unchanged over a wide temperature range.

5. Conclusions

Lasing in vitrified cholesteric materials was studied for the first time. The lasing threshold as well as the number of lasing modes was found to depend crucially on the rate of vitrification, sample thickness, and temperature. Rapid vitrification results in a broad selective reflection band and a multidomain cholesteric structure. Slow vitrification leads to larger domains and a more uniform structure. As a result, lasing in slow vitrified samples has a lower threshold and occurs in narrow wavelength interval.

In monodomain samples the lasing threshold is expected to decrease with the film thickness as a result of increasing quality factor of band edge modes. However, the Wacker cholesteric liquid crystals form relatively large uniform monodomain samples with distinct mode structure at the edge of the selective reflection band when their thickness is equal to or smaller than about 10 μm. In thicker samples, the uniformity of the samples deteriorates and the lasing threshold rises. The lowest threshold was achieved for samples with a thickness of 25–30 μm.

Lasing characteristics were observed to depend strongly on temperature. Above the glass transition, the number of lasing modes is smaller than below, as a result of improved cholesteric texture. The texture is improved by the transformation from the solid to the low viscosity liquid state, facilitating the formation of cholesteric liquid crystals with planar structure. However, the lasing threshold in a liquid state was found to be higher than in the solid state as a result of (i) reduced quantum efficiency of dye molecule emission in the solid and liquid crystalline states, and (ii) reduced *Q*-factor of band edge modes due to the reduced order and lower birefringence of the nematic layers. The factors described here will play a significant role in the lasing of other thermotropic liquid crystalline materials.

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