

**Glassy Liquid Crystals Based on Natural Products  
for High-Peak-Power Laser Optics**

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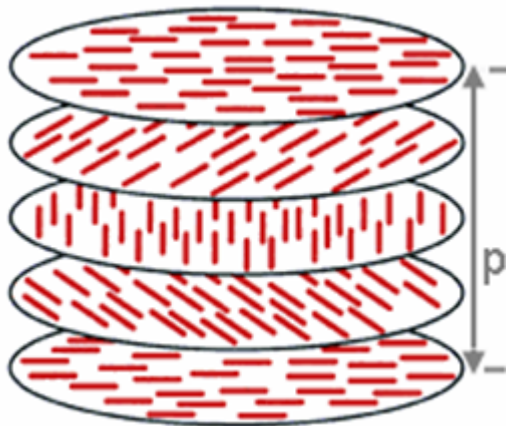
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## **Abstract**

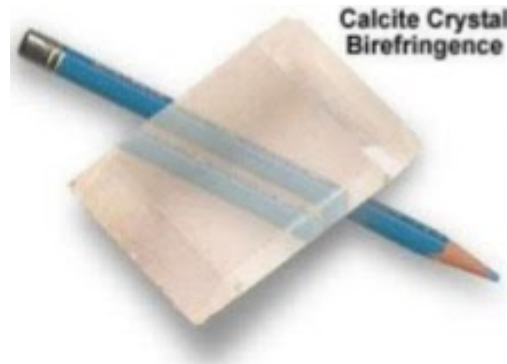
Glassy liquid crystals (GLCs) are of interest for potential application as circular polarizers for high-peak-power lasers such as OMEGA. One new material based on the natural product cholesterol, GLC-Bz3CholC5, is of particular interest because it shows a ten-fold increase in laser-induced damage threshold values as compared to previous GLC materials - a prerequisite for large-scale laser optics applications. The feasibility of using this material for the fabrication of practical, large-area polarization control devices for high-peak-power laser applications was investigated by preparing mixtures of this GLC material with nematic and long-pitch polymer cholesteric liquid crystal (LC) host materials and evaluating their LC phase behavior and optical properties by hot-stage polarizing microscopy. Near-planar alignment of these new materials in assembled devices was achieved by melt-processing and thermal quenching on glass substrates, although significant optical scatter due to LC domain defects remains evident, especially in mixtures containing the long-pitch cholesteric host. A mixture containing 50% GLC-Bz3CholC5 in the nematic host GLC-CChxn3N4 was formulated and used to generate a two-inch-aperture device with few defects, excellent alignment, and blue selective reflection, all of which are promising attributes for future laser applications.

## 1. Introduction

Liquid crystals (LC's) are a form of condensed matter, intermediate in molecular order between a crystalline solid and an isotropic liquid. There are several phases of LC's including the nematic, cholesteric, and several smectic phases. [1] The helical structure of the cholesteric phase and relative ease of generating uniform molecular alignment makes it useful for laser optics applications as circular polarizers. The length of the helical structure in a given cholesteric LC is called the pitch length ( $p$ ) as shown in Fig. 1. Liquid crystals are known for their unique optical and electromagnetic properties, including birefringence and selective reflection. Birefringence, also known as double refraction or optical anisotropy, is an optical effect that occurs when light strikes a birefringent material or surface and diverges into two rays, defined as the ordinary and extraordinary rays. [2] This effect results in double refraction, as illustrated in Fig. 2.



**Figure 1:** Cholesteric LC with pitch length,  $p$ . Cholesteric LC's have the ability to circularly polarize light, a useful optical property in laser systems.



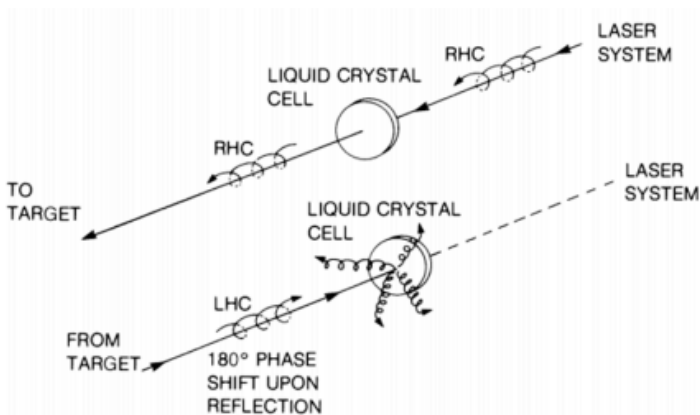
**Figure 2:** Optical birefringence effect in a calcite crystal. In this case, the extraordinary and ordinary waves are separated allowing for the double image of the pencil.

Another optical property of interest of cholesteric LC's is selective reflection, which occurs only for incident wavelengths that satisfy the relationship

$$\lambda = \bar{n}p \quad (1)$$

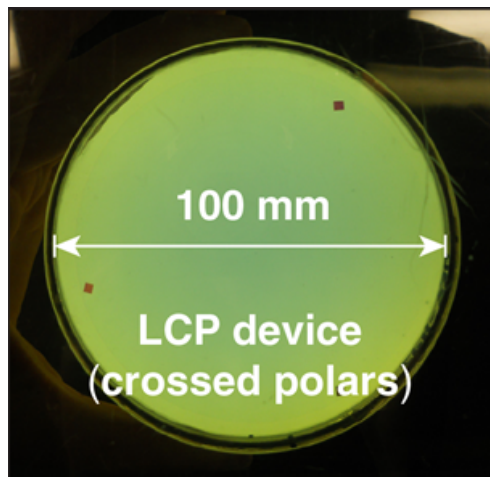
where  $\bar{n}$  is the average refractive index and  $p$  is the helical pitch length. At this point, it is also important to define un-polarized light as light waves vibrating in multiple planes or axes as opposed to polarized light,

which only vibrates in one plane. [3] All natural and artificial light is un-polarized but can be converted to polarized light through the use of linear or circular polarizers. As un-polarized light enters a material exhibiting birefringence and selective reflection, it is separated into two components with opposite handedness (left- or right-handed). One circularly polarized component is reflected from the circular polarizer at its selective reflection wavelength, while the opposite handedness at the selective reflection wavelength is transmitted. All other wavelengths outside of the selective reflection bandwidth (except those that experience intrinsic electronic or vibrational absorbance bands in the LC material) pass through and remain un-attenuated, keeping their polarization states. These optical properties are responsible for generating circularly polarized light, which is useful for maximizing power output and preventing the back-reflection of laser light that can propagate down the laser beam lines and cause damage to optical components, as illustrated in Fig. 3. [1,4] If a laser system propagates right-handed circular polarized light (RHC), as shown in Fig. 3, its right-handed component will pass through the LC device and strike the target; any laser energy that is not absorbed by the target will undergo a  $180^\circ$  phase shift (with a resultant change in handedness) and be reflected from the surface of the target as left-handed circularly polarized light (LHC). This LHC light is unable to pass through the LC device and back to the laser system, and is reflected diffusely from the LC circular polarizer, which reduces the intensity of the reflected light significantly.



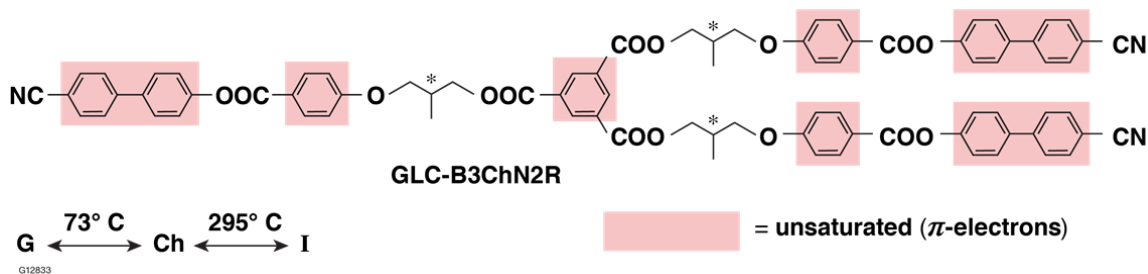
**Figure 3:** Illustration of the use of LC devices to prevent laser damage by changing the handedness of incident light when reflected back from the target. [1]

Current LC laser optics applications employ a low-molar-mass (LMM) LC fluid to produce devices with high optical quality and contrast. The device shown in Fig. 4 demonstrates uniformity in both selective reflection and texture. LMM LC fluids also offer large-aperture device scalability (up to 200 mm), and excellent laser-induced damage threshold (LIDT) values of  $>30 \text{ J/cm}^2$  at 1053 nm and 1 ns pulse width. These LC devices have been used since 1985 on the OMEGA laser system for many device applications including circular polarizers and wave plates. [1]



**Figure 4:** A large aperture, LMM LC fluid device viewed under crossed polarizers. Devices such as these are currently in use on OMEGA.

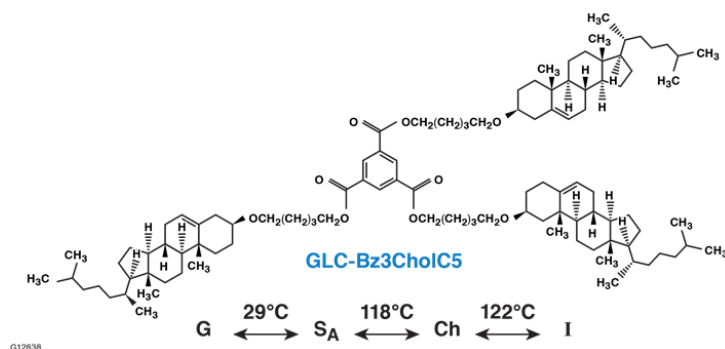
Glassy liquid crystals (GLCs) are a special type of LC that offer the unique optical properties of LMM LC fluids in the form of a solid film below the material's glass transition temperature  $T_g$ . They also offer significant advantages by eliminating LC fluid leakage and thick substrate requirements and may ultimately enable single-substrate devices.



**Figure 5:** A molecular diagram of a first-generation GLC material with ten aromatic rings containing delocalized  $\pi$ -electrons due to the presence of carbon-carbon double bonds.

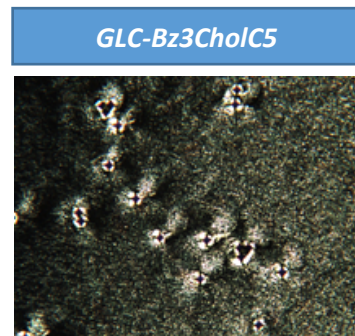
Previously synthesized “first generation” GLC materials contain large numbers of aromatic or unsaturated carbon rings as shown in Fig. 5. [5] This  $\pi$ -electron delocalization has been shown to favor formation of nematic and cholesteric LC phases, stabilize  $T_g$ , and enhance birefringence, all of which are benefits of this GLC. However, these materials have also shown significantly lower LIDT values

( $\leq 1 \text{ J/cm}^2$  at 1053 nm and 1 ns pulse width) than their LMM LC counterparts. [6] The work reported here focused on developing viable GLC mixtures using a second-generation GLC compound (GLC-Bz3CholC5) based on cholesterol. As shown in Fig. 6, this material has an unsaturated benzene core, with three saturated cholesterol pendants and only one isolated double bond in each pendant. In order to fabricate circular polarizer devices from GLC materials, the cholesteric structure observed between 118 and 122° C must be “trapped” by cooling the device rapidly (i.e., “quenching”) to below its  $T_g$  (29° C).



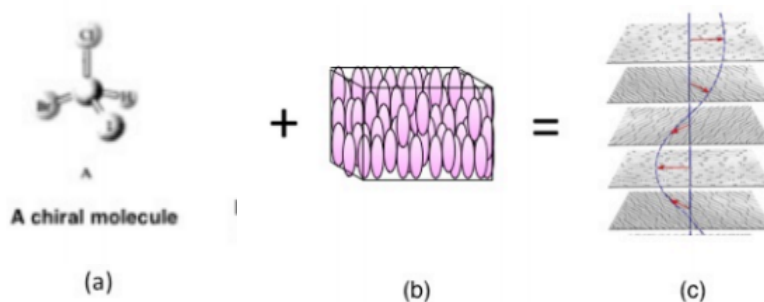
**Figure 6:** A second-generation GLC compound GLC-Bz3CholC5. This material has a significantly higher saturation level, which is beneficial for laser damage resistance, and a wide LC temperature range ( $T_g$  at 29°C, smectic A from 29 - 118°C, cholesteric from 118-122°C, and isotropic above 122°C). The presence of the smectic A phase makes the material more difficult to work with.

Previous work with GLC-Bz3CholC5 has shown that it favors the homeotropic orientation [5] (i.e., the GLC molecules align perpendicular to the glass substrates they are placed on instead of parallel to the substrate, which is required in order to observe selective reflection). This tendency toward homeotropic alignment is due to the presence of the smectic A phase, which adopts this orientation naturally. Another consequence of this underlying smectic A phase is that it produces significant optical scattering effects as the device is cooled rapidly from the cholesteric phase to room temperature (Fig. 7). These optical non-uniformities not only compromise consistent absorbance and transmittance measurements but also hamper device performance in many laser optics applications.



**Figure 7:** Scattering effects in an LC cell made with GLC-Bz3CholC5.

Several mixtures with different GLC nematic or long-pitch cholesteric hosts were used in device fabrication and illustrate that GLCs with natural products are capable of creating well-aligned LC devices for future use in laser optics applications. Figure 8 shows a generalized schematic of the components of the GLC mixtures evaluated. A chiral molecule (e.g., GLC-Bz3CholC5) and a glassy nematic host combine to create a cholesteric GLC with the ability to reflect (and transmit) one handedness of circularly polarized light and function as a circular polarizer. In addition, a procedure for GLC device fabrication was developed using GLC-Bz3CholC5 combined with an inexpensive, commercially available long-pitch polymer cholesteric liquid crystal (PCLC) as a surrogate for the glassy nematic host.



**Figure 8:** Addition of a chiral molecule (GLC-Bz3CholC5) (a) to a nematic host (b) to form a chiral nematic or cholesteric LC (c) that has the ability to reflect one handedness of light and perform the function of a circular polarizer.

GLC-Bz3CholC5 was utilized in this research because it showed a much higher UV transmission and LIDT than GLC-B3ChN2R (Fig. 5). Tables 1 and 2 summarize this data calculated using the Beer-Lambert Law:

$$A = \epsilon bc \quad (2)$$

where A is absorbance,  $\epsilon$  is the extinction coefficient or molar absorptivity, b is the path length, and c is the concentration of the absorbing species. From the first generation GLC material to the second generation GLC material, nine aromatic rings were replaced with saturated rings leading to a sharp decline in the extinction coefficient. The lower the extinction coefficient, the lower the UV absorption. [5] To use these devices for UV laser applications, a low UV absorption and a high UV transmission are desired. Table 2

indicates an almost ten-fold improvement in LIDT values from the first generation GLC to the second material - a clear effect of eliminating the nine aromatic rings. [6]

GLC Material	Generation	# of Aromatic rings	Solvent	Extinction Coefficient ( $\epsilon$ )
GLC-Bz3ChN2R	1	10	THF	4600
GLC-Bz3CholC5	2	1	THF/Isooctane	220

**Table 1:** Comparison of the extinction coefficients ( $m^2/mole$ ) of two GLC materials from different generations. Replacing 9 aromatic or unsaturated rings with saturated analogs yields a much lower extinction coefficient and thus a higher UV transmission/lower UV absorbance. This substantial difference in  $\epsilon$  is promising for laser applications.

GLC Material 22 $\mu m$ thick sample	LIDT ( $J/cm^2$ )	
	351 nm, 1 ns	1053 nm, 1.4 ns
GLC-Bz3ChN2R	0.61	-
GLC-Bz3CholC5	4.89	38.87

**Table 2:** Comparison of the LIDT of the same two GLC materials shown in Table 1. Replacing 9 aromatic or unsaturated rings with their saturated counterparts yields a much higher LIDT value at the 351 nm wavelength. Very large LIDT values are seen at 1053 nm, comparable to those observed for LMM LC fluids. [6]

## **2. Experimental Procedures**

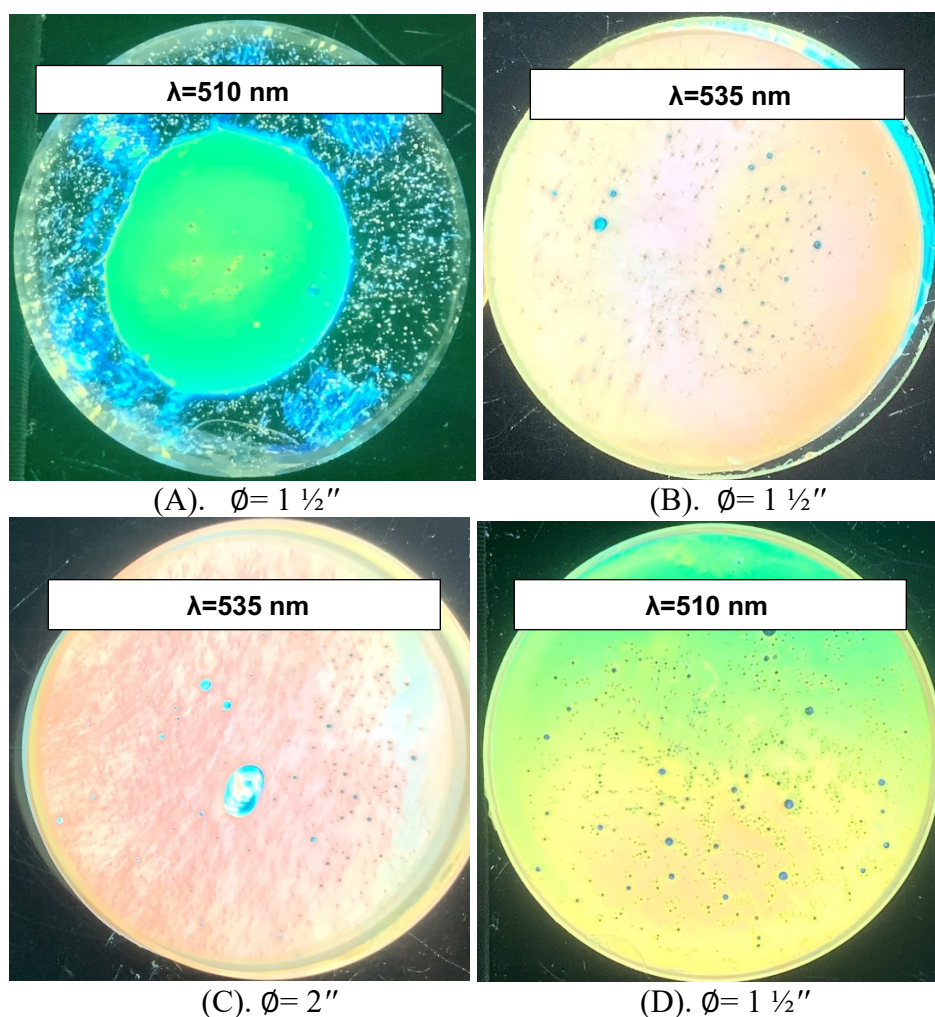
This section describes the development of experimental procedures leading to the fabrication of a GLC device. Section 2.1 describes the creation of surrogate devices used to develop melt-processing techniques. Section 2.2 compares different mixtures and processing techniques used with the surrogate devices of Sec. 2.1. Section 2.3 describes the final processing procedure in detail and Sec. 2.4 describes the formation of the final GLC device.

### **2.1 PCLC Surrogate Device Fabrication**

Two types of commercially available and cost-effective Wacker PCLC polysiloxanes were used to fabricate several surrogate devices with the intention being to demonstrate the basic melt-processing techniques that will ultimately be required to make GLC devices. These PCLC's have very similar chemical



properties but exhibit different selective reflection wavelengths in the blue (510 nm) and green (535 nm). Figure 8 shows several PCLC devices viewed under crossed polarizers. Each of these devices was fabricated using only one type of PCLC. The melt-processing technique used to create each of these devices involves melting the PCLC and applying it onto the central region of a single glass substrate. A portion of the melted PCLC is mixed with a small amount of glass microspheres or fibers, which are then carefully placed into four regions in the shape of a cross to control the cell thickness. The newly applied PCLC is sandwiched between the first substrate and another clean glass substrate and is allowed to cool and solidify (see Fig. 11 in Sec. 2.3: Final Procedure, on Page 14). The two PCLCs used to fabricate these devices have high viscosities and thus trap air bubbles as they are mixed and applied onto the glass substrate, as is evident from the tiny spots in these devices (Fig. 8). Device A was constructed incorrectly by using excessive numbers of glass beads, which resulted in the presence of many microspheres in the empty area surrounding the PCLC in the center of the device. For the other devices, smaller quantities of microspheres were used and limited to only four regions in order to help outline the desired circular shape and prevent excess PCLC material from escaping the device during filling. It is important to note that since microspheres are very small, many microspheres were placed in four specific regions and then allowed to spread out once the device was sandwiched. Ultimately, a high-quality device contains few to no air bubbles, fills the entire substrate with PCLC fluid, and is uniform in color. Because the PCLCs are viscous substances, stirring the materials for an extended period of time further contributes to the air bubbles shown in the devices. In addition, the different shades of the same color in each device show uneven heating and cooling of the PCLC material, which is most clearly visible in Device D.

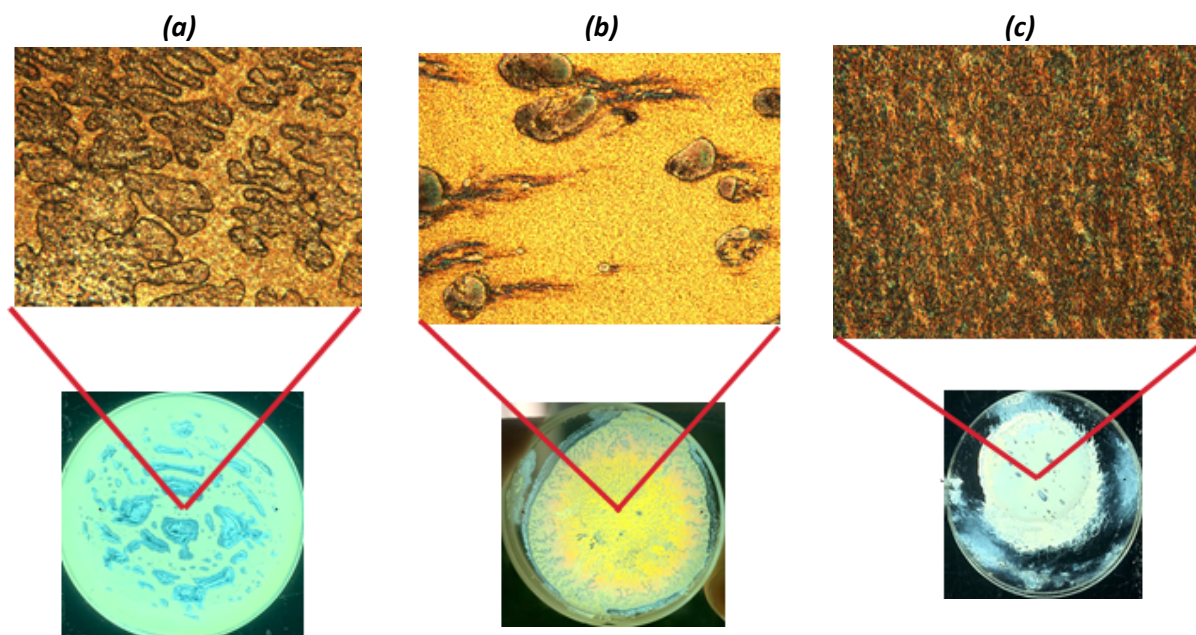


**Figure 8:** Four PCLC surrogate devices and their diameters ( $\phi$ ) shown under crossed polarizers. Devices A and D exhibit a blue color in normal light while B and C display green.

## 2.2 Procedural Development

For procedural development, Wacker LC-4627, another commercially available long pitch PCLC (2200 nm), was used as a surrogate host. The GLC-Bz3CholC5 was used as the chiral substance and combined with LC-4627 in different mass ratios and with different solvents to create several different mixtures (Table 3) that were then used to create devices using the melt processing technique described in Sec. 2.1. Because different ratios of GLC's and PCLC's were employed in the development of the devices, the procedure for each mixture (especially the preparation phase) was different and specific. Two different solvents (dichloromethane and chloroform) were used to dissolve the GLC materials for more efficient mixing and

to allow microfiltration for elimination of extrinsic particulate contamination. Evaporation of the solvents was accomplished in a fume hood using a nitrogen stream, followed by high-vacuum drying using a LabConCo freeze dryer. These samples showed a grainy texture with extensive scattering and as such would not be useful for GLC laser optics applications. As in Sec. 2.1, they were mainly used to develop a complete procedure for device fabrication.



**Figure 9:** Three separate devices (bottom) viewed under crossed polarizers; above each device is shown a photomicrograph of a specific area in that device viewed under crossed polarizers using a polarizing microscope.

Mix #	% GLC-Bz3CholC5 in host	Solvent	Melt Temp (°C)	Processing
1	50	Chloroform (CHCl <sub>3</sub> )	180	1). Anneal 2 hours at 180° C
2	25	Dichloromethane (CH <sub>2</sub> Cl <sub>2</sub> )	170	1). Quench to 25° C
3	25	Chloroform (CHCl <sub>3</sub> )	170	1). Anneal to 155° C 2). Quench to 25° C

**Table 3:** The three different mixtures used to fabricate the devices shown in Fig. 9 along with their processing temperatures and how they were cooled. Quenching is an immediate cooling method in which the device is placed on a cool metal block; annealing is where the sample is cooled slowly to the designated temperature and held there for a prolonged period.

Figure 9 shows that small variations in processing techniques for the three different mixtures shown in Table 3 can make a substantial difference in the final device. [7] For example, Mix #'s 2 and 3 were both processed at the same temperature and contained the same 25% GLC-Bz3CholC5 in the host; however, after dissolving them in different solvents (dichloromethane and chloroform, respectively) and altering the device processing technique by slow cooling of Mix 3 to 155<sup>o</sup> C and then quenching the device (instead of immediately quenching as was done for Mix 2), the two devices showed distinctly different textures under the optical microscope. While Device 2 has several imperfections and a small grainy texture, Device 3 has a more uniform but larger grainy texture with few to no imperfections. The grainy texture in all three devices produced a high degree of optical scattering that prevented any conclusive birefringence or selective reflection testing. All processing temperatures were determined experimentally using hot stage polarizing microscopy, in which small amounts of each mixture were melted to determine their clearing point and glass transition temperature. This procedure allowed determination of viable temperatures for processing each unique mixture using a minimum amount of GLC material.

### **2.3 Final Procedure**

The final procedure consists of three main phases: (1) preparation of the two glass substrates; (2) mixture preparation and filtration; and (3) device assembly using melt processing. On average, it takes roughly two to three days to complete all three phases and assemble a GLC cell. During substrate preparation, two clean glass substrates with the same diameter are selected and cleaned thoroughly using deionized water, detergent, and an ultrasonic cleaner. Next, the cleanest side of each substrate is coated with a polyimide alignment coating, which helps promote parallel alignment of the GLC material. Finally, the coating is buffed in order to promote more uniform alignment. When fabricating an optical device, the GLC molecular alignment needs to be parallel to the substrate in order to achieve the highest quality device. The next process phase was to prepare a uniform mixture by dissolving the GLC nematic (or long-pitch PCLC) and GLC-Bz3CholC5 into either dichloromethane or chloroform to ensure uniform mixing of the individual components. Next, the solution was filtered several times through a sub-micron particle filter

and dried using a simple gas evaporation apparatus (Fig. 10) followed by high vacuum drying overnight. The final phase was device assembly, which consisted of melt processing followed by quenching (rapid cooling), annealing (slow cooling), or a combination of both.

### 1). Substrate Preparation

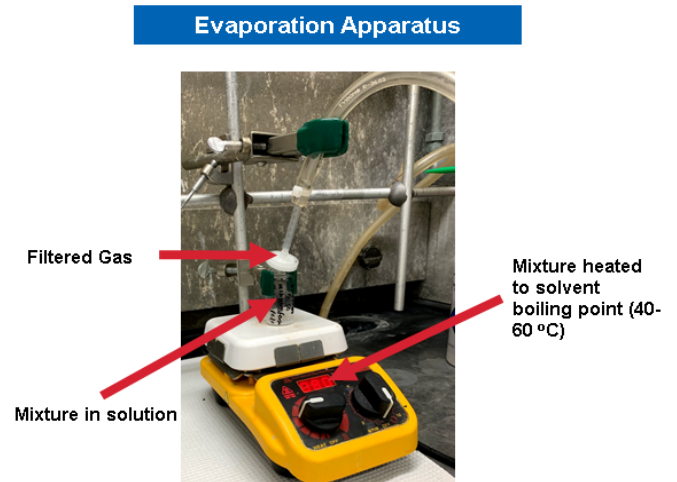
- Wash substrates in an ultrasonic cleaner using a detergent and deionized (DI) H<sub>2</sub>O
- Rinse substrates with DI H<sub>2</sub>O
- Dry substrates in air for several hours
- Spin-deposit polyimide alignment coating onto the substrates and bake at elevated temperature
- Buff the alignment coating using a buffing machine to establish the LC alignment direction

### 2). Mixture Preparation

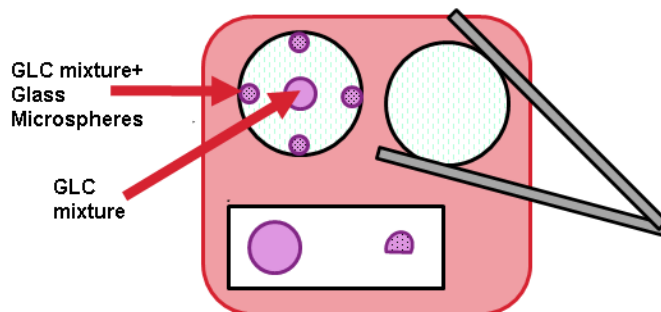
- Dissolve the GLC and PCLC in either CH<sub>2</sub>Cl<sub>2</sub> or CHCl<sub>3</sub>
- Filter through an 0.45 μm particle filter
- Evaporate solvent using N<sub>2</sub> or Ar (see Fig. 10)
- Dry using a high-vacuum freeze dryer

### 3). Melt-Processing (Fig. 11)

- Set hot plate to 10-15° C above T<sub>c</sub> (clearing temperature)
- Melt mixture onto microscope slide and stir until clear
- Transfer the mixture onto the middle of a single substrate, making sure not to touch the substrate
- Mix the remaining clear mixture with glass microspheres and transfer onto 4 corners of the same substrate
- Carefully sandwich the GLC material with the second substrate's coated surface facing the GLC material
- Using previous observations from hot stage polarizing microscopy, decide which cooling method to use
  - **quenching**: quickly transfer the device onto a cool metal surface
  - **annealing**: set hot plate timer to desired time and temperature and monitor cooling



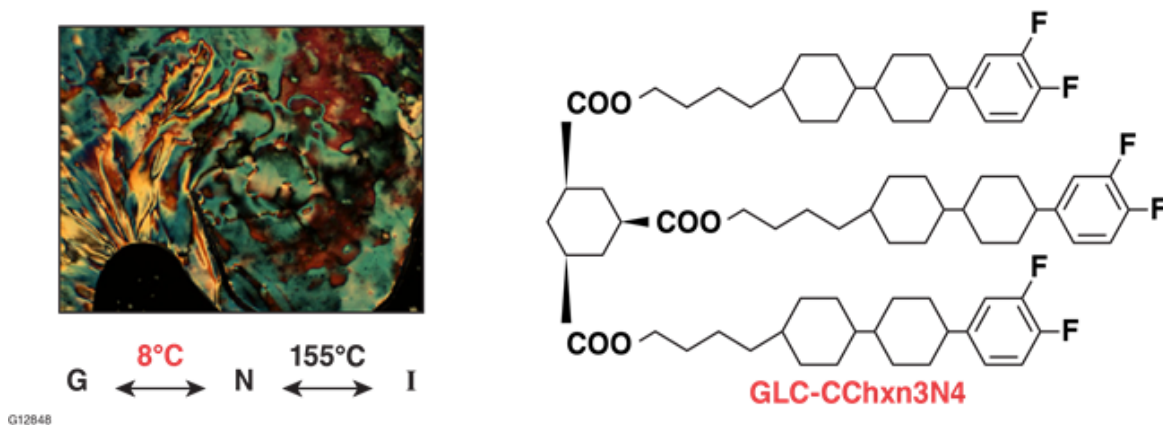
**Figure 10:** The evaporation apparatus used to thoroughly evaporate the solvent from the mixture by running nitrogen or argon gas over it.



**Figure 11:** Schematic showing the basic melt processing technique, which takes place on a hot plate.

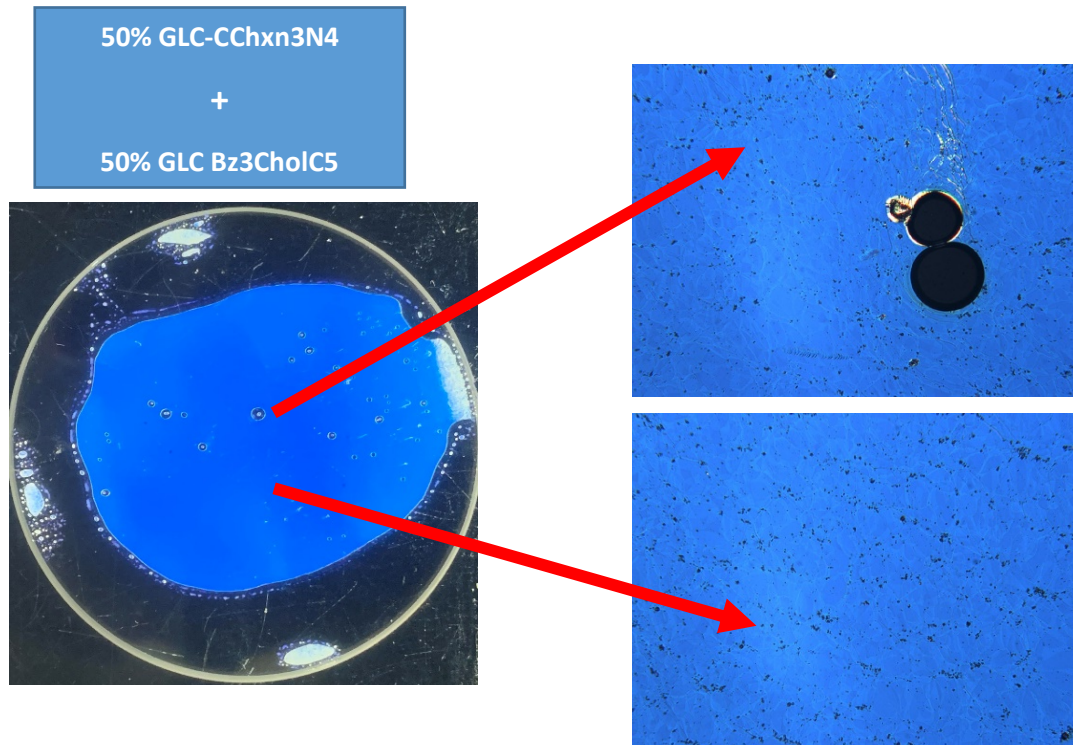
## 2.4 Final Device Development

After the procedure described in Sec 2.3 was developed, a new nematic host, GLC-CChxn3N4 that had been synthesized in very small quantities was mixed with GLC-Bz3CholC5 to fabricate a mixture and device employing only GLC materials. Figure 12 shows the nematic properties and chemical composition of the new host. Nematic LCs possess high orientational order and low positional order; they have many of the optical properties of a crystalline solid, but because they are fluids while in the nematic phase they cannot support a shear stress. When combined with a chiral “dopant”, the resultant mixture inherits the chirality of the dopant, along with associated optical properties such as selective reflection.



**Figure 12:** Nematic texture of the new GLC host viewed under crossed polarizers in transmission using a polarizing optical microscope (left) and its chemical structure (right). The nematic phase has a broad temperature range of 8° to 155°C. The compound has a cyclohexane core with pendants that contain fluorine atoms (F).[6]

The advantage of this host is that its fluorine-containing groups suppress the formation of smectic phases when mixed with GLC-Bz3CholC5. Other advantages are that it (1) allows for a wide temperature range for the mixture's cholesteric phase; (2) induces parallel alignment (see Sec. 1), thus counteracting the natural tendency of GLC-Bz3CholC5 to align homeotropically; and (3) reduces the overall mixture viscosity, which results in fewer bubbles into the device and makes device fabrication much simpler. The new mixture (50% by weight of GLC-Bz3CholC5 in the nematic host GLC-CChxn3N4) was processed at 160°C followed by slowly cooling to 120°C and then the top substrate was moved repeatedly back and forth in the buffing direction to promote homogenous alignment. The device was allowed to cool slowly to room temperature overnight. Figure 13 shows the device under crossed polarizers and regions of the device viewed under crossed polarizers using a polarizing optical microscope. The device has a clear blue selective reflection and few defects (seen in black). The much tinier black specks distributed within the device (right) are most likely trapped air bubbles, which can be eliminated with further processing improvements.



**Figure 13:** Left: the new GLC circular polarizer device viewed under crossed polarizers. Right: Two regions of the device viewed under the polarizing optical microscope. The device is well aligned (as indicated by its uniform blue selective reflection) and shows few major defects (particulates and large air bubbles). The microscopic black objects are air bubbles introduced during shearing of the substrates. The device was fabricated using two thin fused silica substrates with a 2" radius.

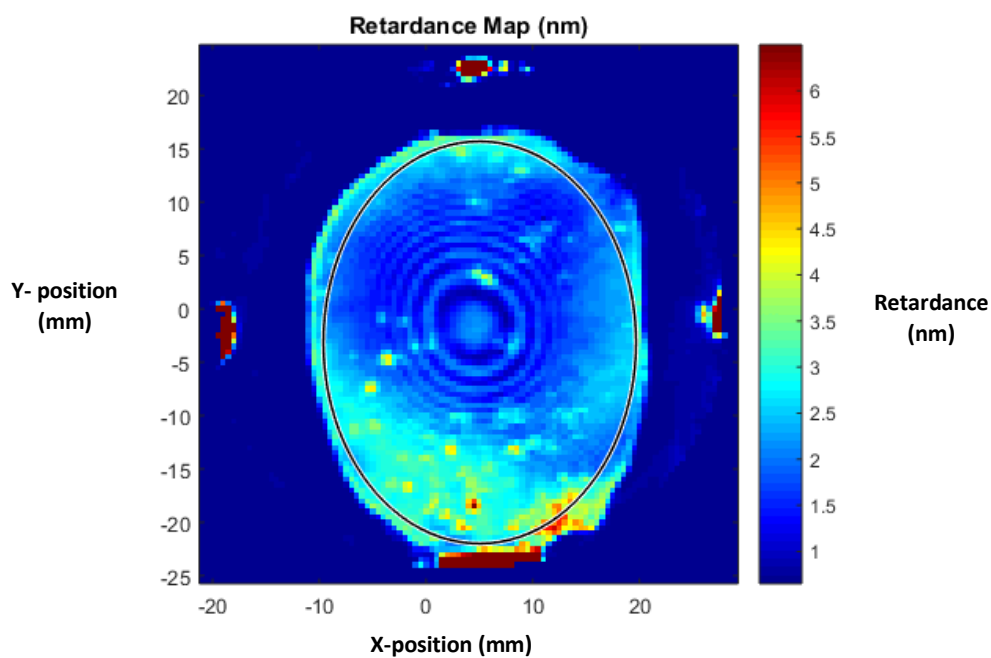
### **3. Results and Discussion:**

Several tests and measurements were conducted on the device shown in Fig 13 to determine the optical quality and properties of the device [8]. If a linearly polarized incident light beam passes through a birefringent material, two polarized components exit the material with a phase difference called the linear retardance. The retardance map (Fig. 14), the optical rotation map (Fig. 15), and the polarization map (Fig. 16), all obtained using a Hinds Mueller Matrix polarimeter, indicate very good LC alignment and little optical scattering. Measurements of linear retardance, optical rotation, and output polarization were taken within the elliptical area of the device where there was GLC material present, as shown in Figs. 14-16. In these three maps, “x-position” and “y-position” indicate the value of retardance, optical rotation, and polarization measured at specified horizontal and vertical distances (in millimeters) from the center of the sample. Because the mixture used to fabricate the device contains chiral material, one would expect a small

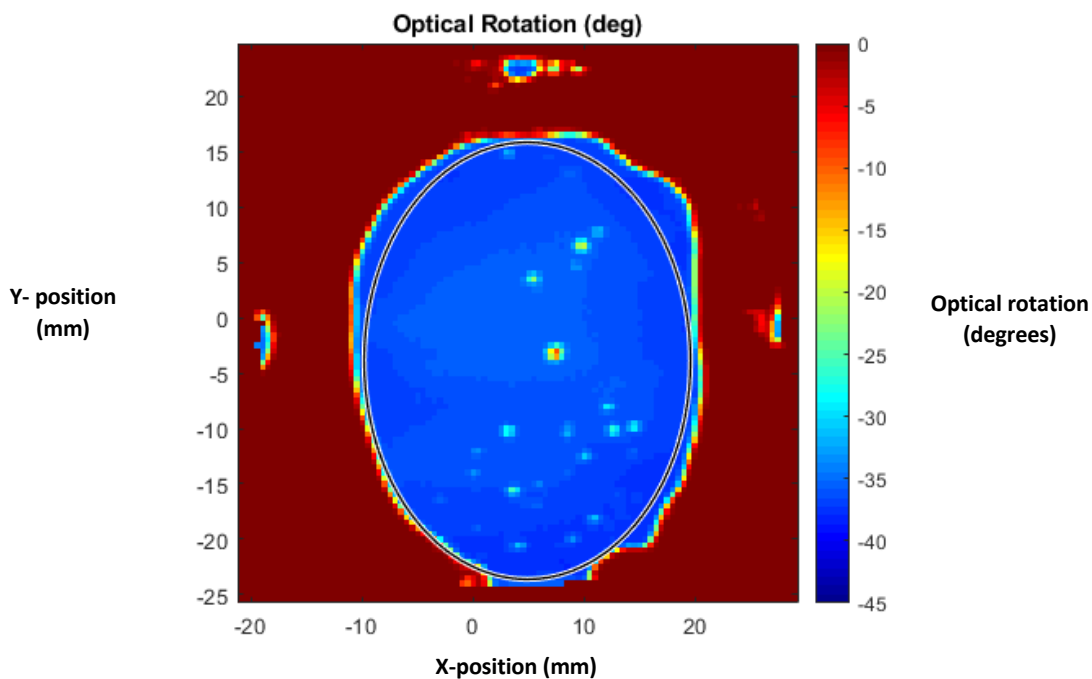


value of linear retardance and largely consistent optical rotation values, both of which are clearly evident (Figs. 14 and 15, respectively). The average linear retardance is  $2.15 \pm 0.65$  nm, with one small area of higher retardance (6.5 nm) shown as a small red spot in the lower region of the outlined device. The retardance map's upper region shows generally lower linear retardance values than its lower region. These results show that there is some residual linear polarization in the device, which must be addressed in future mixtures and device fabrication.

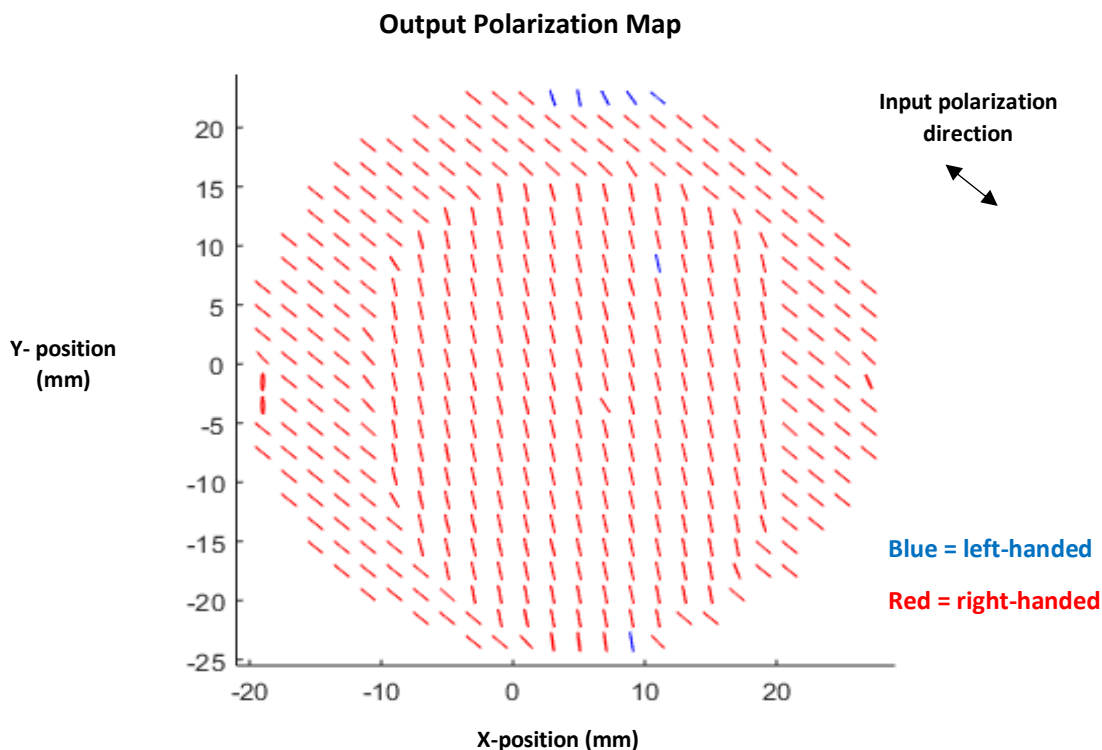
Figure 15 shows the optical rotation of linearly polarized light as it passes through the device. The optical rotation values measured prove that the device is indeed chiral and optically active. Chiral LC materials separate circularly polarized light into two distinct components of opposite handedness (left or right). The output polarization map (Fig. 16) plots the directionality (handedness) of circular polarization. Right-handed circularly polarized light passes through the device while left-handed circularly polarized light is rejected and only present in a few outer regions of the device. The inner region (vertical lines) in Fig. 16 reveals the output polarization rotation produced by chiral GLC material; the outer region ( $45^\circ$  lines) shows the rotation of the incident light polarization passing through the empty regions of the device (essentially identical to the input polarization direction, shown as a black double-headed arrow in Fig. 16). The polarization rotation imparted by the chirality of the GLC is the difference in optical rotation angles between the two sets of lines, which is  $(-)$   $36.2^\circ$ . This result is significant in that it shows that the device has the desired optical properties to be used as a circular polarizer in that it effectively allows only right-handed circularly polarized light to pass through it.



**Figure 14:** Retardance map of the device with an average linear retardance of  $2.15 \pm 0.65$  nm [8].



**Figure 15:** Optical rotation map, indicating that the chiral material in the device is rotating light by a consistent amount of  $(-) 36.2 \pm 1.3^\circ$ . [8]



**Figure 16:** Output polarization map giving the direction of rotation of the incident light after it passes through the GLC device. [8]

#### **4. Conclusions**

Glassy liquid crystals (GLCs) with saturated natural product pendants such as cholesterol can be used effectively to create well-aligned circular polarizer cells with few imperfections, high optical quality and uniformity, high damage thresholds, and excellent alignment. The procedure developed in this work will provide the basis for ongoing GLC mixture development and device fabrication efforts. A 50:50 mixture of GLC-Bz3CholC5 and GLC-CChxn3N4 was developed that showed promising alignment and optical qualities. Processing conditions for the mixture were determined experimentally using hot stage polarizing microscopy; in this manner, phase transition temperatures and alignment quality for different mixture compositions could be observed using very small amounts of material and this information used to determine processing conditions for the fabrication of well-aligned devices. Results showed some desired

optical properties of these GLC materials, but there is certainly more work to be done in eliminating residual linear polarization and scattering effects, as well as microscopic extrinsic particles and trapped atmospheric gases.

In the future, additional mixtures will be developed to reduce scattering and improve GLC alignment. Different alignment techniques such as photo-alignment coatings will be evaluated during device fabrication. Finally, new GLC's with fully saturated cores and pendants based on other natural products (such as the steroid epiandrosterone) are under development and will undergo similar device fabrication techniques as the ones developed in the procedures described here. If a viable mixture composition is developed, future research will involve the fabrication of single substrate devices, which would be a significant benefit to laser systems such as OMEGA and even the National Ignition Facility.

## **5. Acknowledgments**

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## **6. References**

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