Chapter 3

Fabrication Process, Instruments and Principle

3.1 Introduction

FGLCs as well as FGLC mixtures were prepared in this study, all the fabrication processes and instruments which are available to characterize the parameters of LCs will be introduced in this chapter. The fabrication processed included the FGLC mixtures preparation and FLC devices preparation. Instruments such as differential scanning calorimetry (DSC), polarizing optical microscope (POM), and optical system were utilized to characterize thermal properties, textures, and electro optical characteristics, respectively, the major features of the above mentioned instruments will be illustrated in the chapter.

3.2 Fabrication Process

Two fabrication processes including the preparation of FLC mixture and the measurement steps of LC parameters were described into lists. To prepare FGLC mixtures, FGLC compounds were used as chiral dopant doped in the SmC host W206A. And a well mixed mixture was prepared by dissolve them with dichloromethane. Above all characteristics, thermal properties were first characterized, and the other parameters were characterized after filling FLC material in the pre-made cell.

3.2.1 FGLC Mixture Preparation

a. Measure the desirable weight of FGLC compound and SmC host W206A with an electronic scale and put together in a 10ml vial.

b. Dissolve them with dichloromethane and filter the solution with 0.2μm filter.

c. Cover the vial with tinfoil and poke some holes, than the vial was put on 50 ^{o}C hot

plate, and was purged nitrogen in small flow. Most dichloromethane will evaporate and flow through the holes in this process.

d. Final baking with vacuum oven

3.2.2 Measurement

a. Measure the thermal property with DCS from -30 ^{o}C to 150 ^{o}C at heating and cooling rate 20 ^{o}C /min.

b. Define LC phases under POM, temperature of material is controlled with hot stage.

c. Calculate the cell gap of pre-made EHC cell with UV-Visible at three regions of the cell in ITO area and get the average.

d. Fill LC with the following steps, and the filling and annealing temperature are listed in Tab. 3.1

1. Fill LC at Isotropic.
2. Naturally cool down to chiral nematic mesophase, and stay for 5 minutes.
3. Naturally cool down to chiral smectic C mesophase, and stay for 30minutes.
4. Naturally cool down to room temperature.

Tab. 3.1 Filling temperatures of FGLC mixtures.

Material	***Fill temp. (℃)***	***N* temp. (℃)***	***SmC* (℃)***
2% FGLC-1	110	95	70
4.3% FGLC-3	110	98	80
FGLC-2	150	None	120
R2301	100	80	60

e. Alignment and its ON/OFF textures are observed under POM.

f. Measure the electro-optical characteristics with optical system.

g. Measure spontaneous polarization and other parameter.

3.3 Instruments and Principle

After the FGLC mixtures were prepared, the LC parameters will be characterized, and brief introductions of instruments and principle will be given in this section.

3.3.1 Differential Scanning Calorimetry (DSC)

PerkinElmer Diamond DSC and added liquid nitrogen cooling system were utilized to characterize the thermal properties as shown in Fig. 3.1, and the instrument was adjusted with Indium at heating and cooling rate 20 ^{o}C/min. 3-5 mg FGLC materials was put in the aluminum pan, and was analyzed at heating and cooling rate 20 ^{o}C/min. Phase transition temperatures were defined with the endotherm peaks in the heating curve and exotherm peaks in the cooling curve.

Fig. 3.1 Photo of PerkinElmer diamond DSC and its liquid nitrogen cooling system.

3.3.2 Polarizing Optical Microscope (POM)

Liquid crystal textures were observed under POM, OLYMPUS BX51 as shown in Fig. 3.2, the magnifications of POM are 100X, 200X, 500X and 1000X with changeable object lens of 10X, 20X, 50X and 100X, respectively, and a 10X eyepiece. Two measurable modes depend on transparent and reflective substrates are utilized with bottom light source and top light source, respectively. And adjustable and movable polarizer can be utilized in both modes. Images observed under POM can be captured under CCD, and the parameters such as, distance, area and angle can be calculated with its software.

Fig. 3.2 Instrument photo of POM, OLYMPUS BX51.

3.3.3 Hot Stage

Hot stage of METTLER TOLEDO FP90 was utilized in the experiment with its controllable heating and cooling rate as shown in Fig. 3.3, and the indicate temperature is stable with its accuracy to ±0.4 ^{o}C. The highest temperature is given at 375 ^{o}C and the lowest temperature is regarded as room temperature, and benzoic acid is used to adjust this instrument. Hot Stage can also be utilized in optical system or POM with the small transparent quartz pin hole.

Fig. 3.3 Instrument photos of center controller (left one) and hot stage (right one)

3.3.4 UV-VIS Spectroscopy

UV-VIS Spectroscopy, LAMBDA 650 PerKin Elmer as shown in Fig. 3.4 is operated in the ultraviolet visible spectral ranges (190 nm – 900 nm) with the resolution ≦ 0.17 nm, and the spectrometer features a double-beam, double monochromator, ratio recording optical system. The instrument is usable in a wide range of applications as indicated by the performance specifications. Absorption and transmittance of materials can be characterized with LAMBDA 650, and the large cavern supposes the space for polarizers and other expanded object.

Fig. 3.4 Instrument photo of UV-VIS Spectroscopy, LAMBDA 650 PerKin Elmer.

In this thesis, each cell gap of pre-made cells (form EHC) was characterized with spectroscopy by interferometric methods [23] as the formula in (3-1).

$$\text{Cell gap} = (x+1)(\lambda_1 \times \lambda_2) / 4(\lambda_1 - \lambda_2) \quad \text{(unit: nm)}, \tag{3-1}$$

Where x is the peak numbers between two selected peaks and themselves.

λ_1 and λ_2 are the wavelength of selected peaks. $(\lambda_1 > \lambda_2)$

As the interference data of Fig. 3.5, we choose the peaks of 679 nm and 491 nm, and the peak numbers between them and themselves is 5, thus the cell gap of this cell is :

(5+1) (679 × 491) / 4 (679 - 491) = 2660 nm.

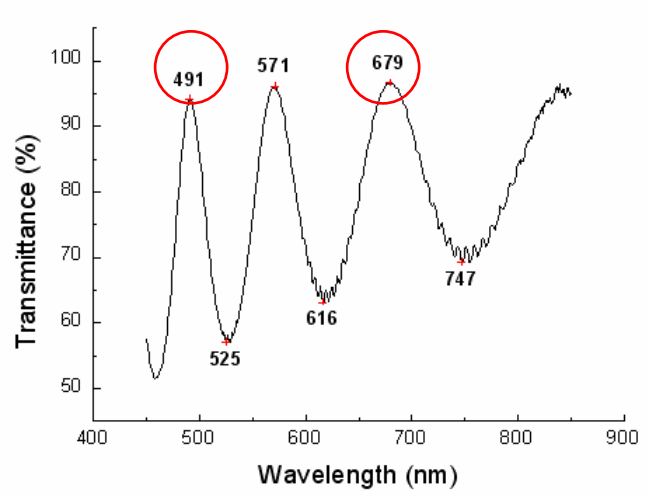

Fig. 3.5 Interference data of one EHC cell by UV-Visible

3.3.5 Optical System

In the optical system as shown in Fig. 3.6, LC cell is put between crossed polarizers, and driven by waveform generator, WFG500 which is connected with computer through GPIB interface. He-Ne laser (λ = 632.8nm) as light source, the optical signals are received with photo detector, and the intensity of laser light is transformed into voltage, and are exhibited with oscilloscope or multimeter. Electro optical characteristics are measured with this system, and the cell can be heated with hot stage to get the temperature dependent data.

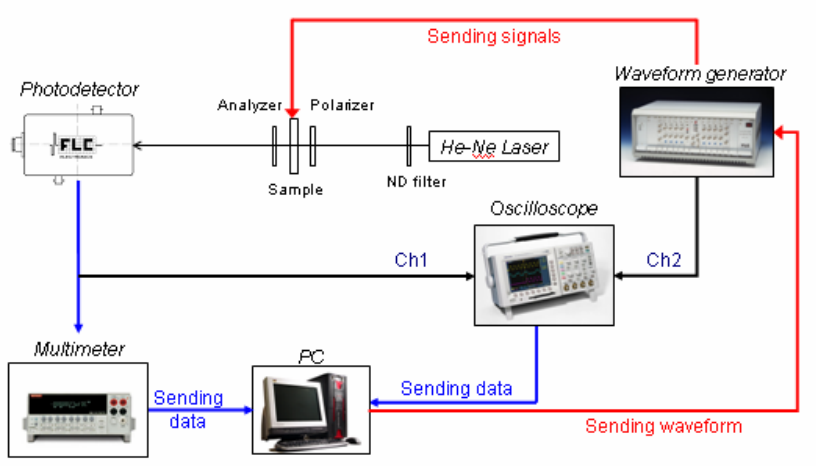

Fig. 3.6 Diagram of optical system.

a. Waveform Generator

Multi-channel high voltage waveform generator WFG500 (made in FLC ELECTRONICS AB) is utilized to applied voltage across LC cells. WFG 500 is powerful to be a high voltage signal generator with 8 output channels and waveform-designed ability. Any waveforms even they are not regular can be designed with the maximum output voltage for ±100V and the minimum designable pulse width for 200ns.

b. Photo detector

Optical signals of laser beam are detected with photo detector, PHOTODECTECTOR PIN 20 (from FLC ELECTRONICS AB), the light sensitive component, silicon PIN photo-diode contribute to a good compromise between sensitivity and speed, and the signal with its response more 40ns can be detected by this instrument.

3.3.6 Measurement of Spontaneous Polarization

Several methods [24-28] have typically carried out to measure the spontaneous polarization, in this thesis, the value of spontaneous polarization was characterized by field reversal method [26], the circuit set up is shown in Fig. 3.7, FLC cell is connected with a 100k resistance in series, by applying a 25Hz triangular wave, current resulting from polarization reversal is characterized, and the current consists of three components as shown below.

$$I = I_c + I_p + I_i = C\frac{dV}{dt} + \frac{dP}{dt} + \frac{V}{R}, \qquad (3.2)$$

Where, I is the total current,

Ic is the current due to charge accumulation,

Ip is the current induced by polarization,

Ii is due to ion flow.

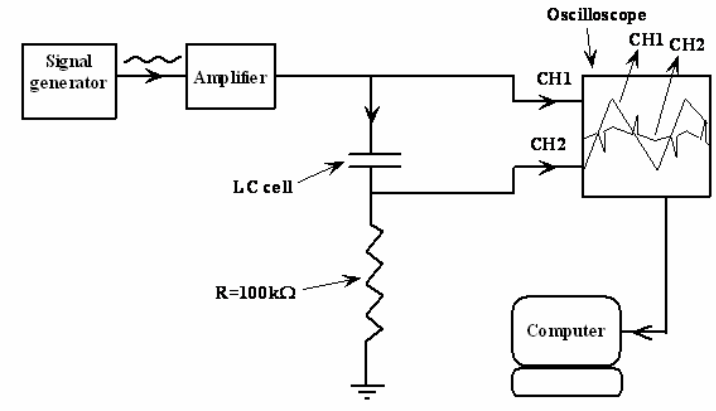

Fig. 3.7 Circuit set-up of measuring spontaneous polarization.

A liquid crystal cell can be regards as a resistor and a capacitor connected in parallel, and P is the charge induced by spontaneous polarization, the value can be calculated by integrating the current of Ip as shown in Fig. 3.8.

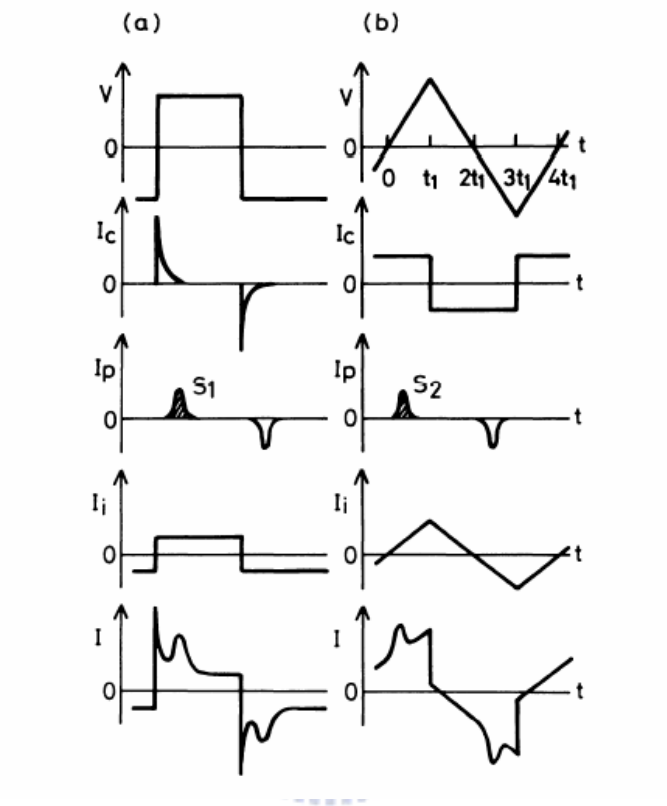

Fig. 3.8 Schematic illustration of the current induced by (a) applying a square wave. (b) applying a triangular wave. Three contributions Ic, Ip, Ii, to the overall current I [26].

Both square wave and triangular wave can produce ferroelectric switching with field reversal, for fast switching materials, however, the capacitive current and polarization reversal currents are often difficult to be separated if the ferroelectric switching speed is not slow enough comparing to the RC time constant of the circuit.

Ion effects as well as spontaneous polarization present the current by field reversal method. By the following methods we can verify that the current peaks are indeed produced by spontaneous polarization or not.

(1) Under the same conditions, heat up the sample into isotropic phase and see whether the peak disappears or not, the peak will disappear if it is due to spontaneous polarization, otherwise, it is due to ions.

(2) Measure the value of polarization at different temperature of SmC* phase, and the Ps value will decrease with the increase of temperature. If it is due to ions, the value will increase with increasing temperature.

(3) Vary the cell gap, if the peak is due to ions, it may not exhibit in thicker cell (over 20 μm).