## Decrease in Measurement Accuracy of Flexoelectric Coefficient by contamination in Liquid Crystal Materials

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#### ABSTRACT

It was confirmed that contamination in liquid crystal (LC) deteriorates the reliability of the estimation of device parameter such as flexoelectric coefficient especially when measuring the phase difference of LC sample by means of the symmetric oblique incident transmission ellipsometry with applying DC voltage.

#### **1** INTRODUCTION

Many reports on flexoelectric effect have been published recently. Since the deterioration of image quality of liquid crystal display (LCD) due to flexoelectric effect has been pointed out, there has been much interest in methods for evaluating flexoelectric effect of nematic liquid crystal (NLC) [1]. Up to today, several kinds of measurement method have been proposed [2]. A series of our papers on a method of determining flexoelectric coefficients [3] are based on the concept proposed by Takahashi *et al.* [4], where we adopted our original transmission ellipsometry which has been developed for the purpose of determining the surface anchoring energy between NLC and surface alignment films [5].

Needless to say, proper samples should be prepared to make reliable measurements of device parameters of NLC. There are many phenomena that cause measurement deviation. Especially for the measurement of flexoelectric coefficient, because of its polar response on applied DC voltage, the effect of ambient component such as surface alignment film has been considered [6]. For the purpose of reducing the measurement deviation and establishing a measurement method of flexoelectric coefficient, a series of measurement procedure was proposed [3]. Namely, one sample cell holds different types of compartments in which hybrid alignment (HAN) or uniform alignment are



Fig.1. Schematic of the sample HAN cell.

prepared and are driven by the vertical or in-plane electric voltage. As a result, cell gap and surface anchoring energy can be determined by the precedence measurement with the uniformly aligned compartment, and then only flexoelectric coefficient can be determined by the HAN compartment in the subsequent experiment. In this paper, however, still we found that the reproducibility of the data was insufficient, and possible cause such as contamination is discussed.

#### 2 **EXPERIMENT**

#### 2.1 LCD Sample Preparation

We choose HAN alignment to determine a sum of flexoelectric coefficient  $(e_{11}+e_{33})$  because HAN alignment possesses splay and bend deformation, where  $e_{11}$  and  $e_{33}$  are for splay and bend deformation, respectively. Sandwich typed NLC cell with indium tin oxide (ITO) thin-film as a transparent electrode is prepared as shown in Fig. 1. To produce HAN region, upper glass substrate surface was covered by a prerubbed polyimide for planar alignment, and lower glass substrate was covered by another type of polyimide for vertical alignment without rubbing treatment. The bead spacer was sandwiched by two glass substrates, the nominal cell gap was 10 µm. Two



Fig.2 Photograph of sample cell under the crossed polarizer films. (a) Fixed by foldback clip.(b) Cemented by epoxy resins. The white arrow corresponds the axis of polarizer, and the length of the arrow exhibits 15 mm.

glass substrates were fixed by foldback clip or cemented by epoxy resins. Figure 2 shows the aspect of the fabricated sample cells after filling the LC materials, MLC-6608 (Merck). Table 1 shows the birefringence  $n_e$  and  $n_o$ , elastic constant for splay and bend deformation, and dielectric constant  $\varepsilon_{\parallel}$  and  $\varepsilon_{\perp}$  of MLC-6608.

Table 1 Physical parameter of MLC-6608 at 25 °C		
Birefringence	n <sub>e</sub>	1.5586
@589nm	$n_o$	1.4756
Elastic constant	K <sub>11</sub>	16.7
[pN]	K <sub>33</sub>	18.1
Dielectric	$\mathcal{E}_{\parallel}$	3.6
Constant @1KHz	$\varepsilon_{\perp}$	7.7



# Fig.3 Transmission Ellipsometry with LC sample rotation stage.

#### 2.2 Transmission Ellipsometry

The measurement system of the transmission ellipsometry composed of the polarizer, analyzer and photo-elastic modulator (PEM) is illustrated in Fig. 3. The slow-axis of PEM is parallel to y-axis, and the sample cell is placed between PEM and analyzer. The sample on the rotating stage can rotate ±45 degrees around the y-axis in order to carry out symmetric oblique incident transmission ellipsometry (SOITE) [5]. The incident angle  $\beta$  is set ±45 degrees and wavelength of the diode-pumped solid-state laser as a light source  $\lambda$  = 589 nm. Phase difference  $\Delta$  for + $\beta$  incident and - $\beta$  incident ( $\Delta^+$  and  $\Delta^-$ ) as a function of the applied voltage is measured with this transmission ellipsometry system, and the flexoelectric coefficient is estimated by fitting the difference  $\Delta^- - \Delta^+$  to the theoretical value. To estimate the cell gap and surface polar anchoring energy by means of the precedence  $\Delta^-$  –  $\Delta^+$  measurement, AC rectangular wave voltage of 1 kHz was applied. To estimate e11+e33 by means of the subsequent  $\Delta^- - \Delta^+$  measurement, DC voltage was applied for  $\Delta$  measurement, where an appropriate interval is set between the sinusoidal applied voltage in order to avoid the uneven accumulation of ions. Figure 4 shows the devised wave form to estimate  $e_{11}+e_{33}$ .



### 1 Period (4 s)

Fig.4 Timing chart of applied voltage and  $\Delta$  measurement, where 1 period is 4 s.

#### 3 RESULTS and DISCUSSION

Figure 5 shows typical results of the AC applied voltage dependence of  $\Delta^- - \Delta^+$ . Here, let us allow to emphasize that the sample was cemented by using epoxy resins after filling the NLC. In order to confirm the reproducibility of our measurement,  $\Delta$  measurements were done immediately after NLC injection, 24 hours later, 48 hours later, and 120 hours later, respectively. The measurement deviation of the experimental data over time seems to be tolerably small. Usually, from this experiment using planarly aligned compartment neighboring HAN compartment, first we can determine the cell gap and surface polar anchoring energy of the whole LC sample cell which are necessary value to estimate e11+e33 by numerical calculation [3]. Note that the cell gap and surface polar anchoring energy determined in one section of same sample seems to be same to that in neighbor HAN alignment region.





Figure 6 represents the results of the DC applied voltage dependence of  $\Delta^- - \Delta^+$ , also  $\Delta$  measurements were done immediately after NLC injection, 24 hours later, 48 hours later, and 120 hours later, respectively.

The sample cell was cemented with using epoxy resins. From this experimental result, it was found that the shape of  $\Delta^- - \Delta^+$  fluctuated every time irregularly when the experiment was performed. The estimated e11+e33 varied from -33 to 49 pC/m. In the early days of this experiment, this fluctuation and resultant deviation of flexoelectric coefficient were thought to be due to the impurities in NLC materials. In a past literature, Takahashi pointed out that avoiding the influence of impurity ions contained in LC materials is difficult when a characteristic of the LC materials is obtained under applying the DC voltage [7]. However, it is natural to consider that the NLC material is synthesized with quite high purity and that impurities are mixed in during the cell fabrication process. Above all, epoxy resins sealant that directly touches NLC is most suspicious component.



Fig.6 Time dependence of  $\Delta^- - \Delta^+$  measurement under DC applied voltage.

То verify hypothesis, similar  $\Delta^- - \Delta^+$ this measurements under DC applied voltage were performed using unsealed samples (namely sample appearance is of Fig.2(a)), as shown in Fig. 7. It can be seen that the measurement deviation of  $\Delta^- - \Delta^+$  over time is relatively small after 24 hours compared with Fig.6. From this experimental result, it was found that the deviation can be suppressed by not using the epoxy resin. However, impurities from ambient seems to be contaminated into NLC because no sealing material was cemented. From these experiments, it can be summarized that  $\Delta^- - \Delta^+$ measurement should be performed immediately after NLC injection.

#### 4 CONCLUSIONS

It has been recognized that the sample cell fabrication process and components of the sample cells affect the electro-optical measurement accuracy. In this experiment, it was clearly shown that the measurement deviation which was not remarkable under the AC voltage application was remarkable under the DC voltage application. A series of experiments show that the measurements of flexoelectric coefficient should be completed before the impurities diffuse into LB bulk.



Fig.7 Time dependence of  $\Delta^- - \Delta^+$  measurement under DC applied voltage, where the sample NLC was unsealed with epoxy resins.

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