X-ray Scattering Studies for Phase Separated Composite Organic Films

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Abstract

The ratio of optimized concentration on optical characteristics for phase-separated composite organic films (PSCOF) liquid crystal display is 30% of pre-polymer (NOA65) and 70% of ferroelectric liquid crystal (Felix). The layer structure in ferroelectric liquid crystal cell made by 30% NOA65 and 70% Felix materials is tilt-bookshelf layer structure. The angle of tilt-bookshelf structure are 17°, 12° which are almost same of tilt angle of ferroelectric liquid crystal in Sm C phase. We know that this result is from compensating the layer buckling. In this paper, we will discuss the effect of layer structure in PSCOF cell on ratio of concentration between pre-polymer and liquid crystal by x-ray measurements. We believe that technology of PSCOF is a good solution to solve the problems of align-defect and mechanical shock for future TV application and plastic LCD.*

1. Introduction

Liquid Crystal Display (LCD) applied for TN, IPS, and PVA mode shows good quality of image, but there is still problem obtaining the moving picture for AV applications. For this reason, the new mode of LCD satisfying wide viewing angle and fast response time is needed to develop by LCD manufacturing companies. The ferroelectric and antiferroelectric liquid crystals [1] have been attracted that they have the big potential of display applications because of fast switching and wide viewing properties. But there are serious problems to be solved for LCD application, such as low contrast ratio caused by zig-zag defects, mechanical durability, and so on. For many years, many LCD companies made great efforts to solve these problems [2-3], but they didn't succeed to solve them for mass production.

It has recently been shown a possibility in order to make LCD panel overcome the above problem by means of a phase-separation technology [3] when FLC is used. It has been reported a method of preparing LC devices using anisotropic phase separation [4], which is referred to as phase-separated composite organic films (PSCOFs). It is shined by ultraviolet (UV) light onto one side of a cell filled a binary mixtures of LC and pre-polymer. Illumination of low energy of UV light and sufficient exposure time can be allowed the complete separation of LC and polymer.

In this paper we briefly report some x-ray experimental results of layer structure in cell on different concentration of binary mixture. And, we analyze the relationship between chevron structure and tilt-bookshelf structure at the PSCOF technology.

2. Experimental Details

The technique used to construct the PSCOF cell is shown in Fig. 1 whose technique is similar as that used for making polymer-dispersed liquid crystal (PDLC) devices. A pair of substrates coated with transparent electrodes of indium-tin-oxide (ITO) is prepared to make cell. One of the substrates is coated by a commercial polyimide, RN1286, and then is rubbed to enforce for LC alignment. The other substrate is remained. For a binary mixture, we used commercially available photo-curable pre-polymer NOA-65 from Norland and Liquid Crystals Felix-106/100 from Clariant. The ratios of the photo-curable pre-polymer NOA-65 and LC are 20:80, 25:75, and 30:70. Two components are mixed for a few days to make homogeneous binary mixture, and then mixture is filled into the cell by capillary action at a temperature well above the clearing point of the LC. Phase separation is carried out at a temperature above 100 ℃ by exposing UV light to the cell normally on

the no-coated substrate by polyimide. The source of UV light was a high-pressure xenon vapor lamp operated at 200 W of electrical power, and exposure time is 15 min.

After making the PSCOF cell, we tried to make very thin glass cell (bellow 100 m thick cell) by etching used hydrofluoric acid solution to minimize x-ray absorption to the glass.

The phase transition sequences of FLC (Felix-015/100, Felix-016/100) that we used are as followed;

I-(86)-N-(83)-Sm A-(72)-Sm C* for Felix-015/100 and I-(94)-N-(85)-Sm A-(72)-Sm C* for Felix-016/100.

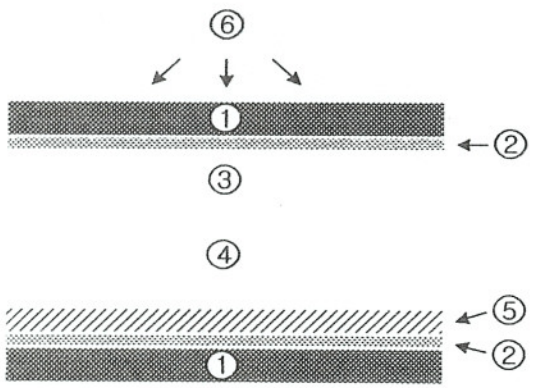

Fig. 1. Schematic of the structure of a PSCOF cell. ① glass substrates, ② electrodes, ③ polymer region, ④ LC region, ⑤ alignment layer, and ⑥ UV source.

3. Results and discussions

The high resolution x-ray scattering experiments were performed by 18kW Rigaku RU-300 rotating anode generator, a two-circle Huber goniometer with a pair of (111) face polished perfect germanium single crystals as monochromator, and analyzer. All measurements were performed with a cooling sequence from isotropic phase to room temperature.

The temperature dependence of the layer spacing of Felix-015/100 ferroelectric liquid crystal is shown in Fig. 2. The layer spacing was calculated from the Bragg's law which is $2d\sin\theta = n\lambda$, where d is the layer spacing and λ is 1.54 Å which is x-ray wavelength.

Figure 3 shows the temperature dependence of the layer tilt angle (the chevron angle) to the normal of the substrates. It was almost same of the molecular tilt angle for compensating the layer buckling, which can be calculated by using the equation, $\theta_{tilt}=\cos^{-1}(d/d_A)$, where d_A is the layer spacing in Sm A phase. The molecular tilt angle obtained by x-ray scattering generally gives rise to the average of tilt angle among all molecules. We can figure out the molecular tilt angle corresponding to chevron angle. Also we can see that the angles of the chevron structure were observed symmetrical to the normal of the substrates.

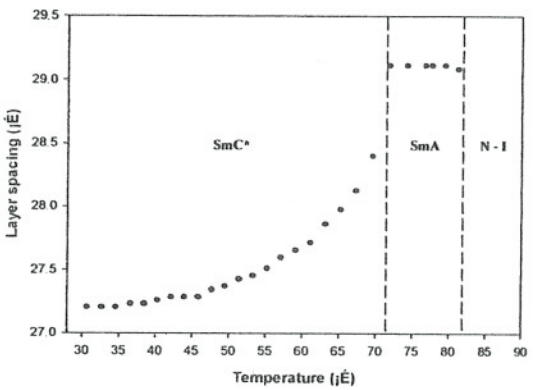

Fig. 2. The layer spacing on temperature for pure Felix-015/100.

Figure 4 and 5 shows a comparison of the layer structure between SSFLC cell made by pure Felix-015/100 and PSCOF cell made by binary mixture. We can see that the chevron layer structure appears in Sm C* phase in SSFLC cell as a usual, which is known from two peaks in the theta scan at 57.6 ℃ (Fig. 4). The peak position is symmetric each other, but the intensity of left peak and right peak is shown as an asymmetry. It means that the layer tilt angle has a symmetrical position to the surface normal, but a different domain length of Chevron structure.

Figure 5 is obtained by theta scan at 57.6 ℃ using PSCOF cell made by binary mixture of 70wt% of the FLC Felix-015/100 and 30wt% of pre-polymer NOA65. There is only one peak in theta scan. The strong peak position is near 16° which means the layer structure of PSCOF cell is a tilt-bookshelf structure and layer structure is very uniform. The tilt angle of bookshelf structure is same as layer tilt angle as shown in Fig. 3 and Fig. 5 because of compensating the layer buckling. We can expect that the PSCOF technology is one solution to naturally remove the zig-zag defect.

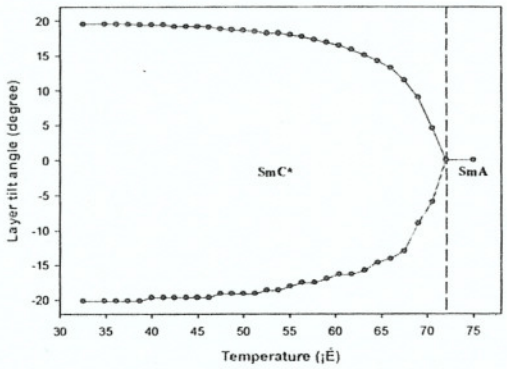

Fig. 3. The temperature dependence of the layer tilt angle for pure Felix-015/100. In Sm C* phase, the layer tilt angle is symmetric.

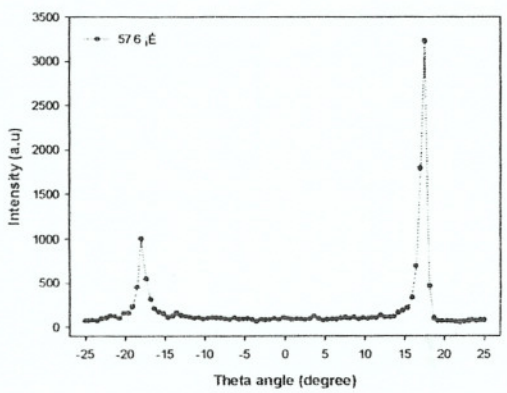

Fig. 4. The theta scan of pure Felix-015/100 at 57.6 ℃. The peak positions are symmetric but the intensity of the peaks is not symmetric.

Next, we want to compare the x-ray scattering results obtained by pure Felix-016/100 cell, and by PSCOF cell, 70wt% of Felix-016/100 FLC and 30wt% of pre-polymer NOA65.

Figure 6 shows the theta scan for pure Felix-016/100 cell in Sm C* phase at 63.1 ℃ whose peak positions are symmetric, but the intensity of left and right peaks is asymmetry. It looks same result as Felix-015/100 cell.

Figure 7 is also the theta scan of PSCOF cell made by 70wt% of Felix-015/100 FLC and 30wt% of pre-polymer NOA65 at 63.2℃. The peak position is on 12° which is almost same result of layer tilt angle in this cell at this temperature. If temperature decreases, both tilt angles increase as results of Fig. 5.

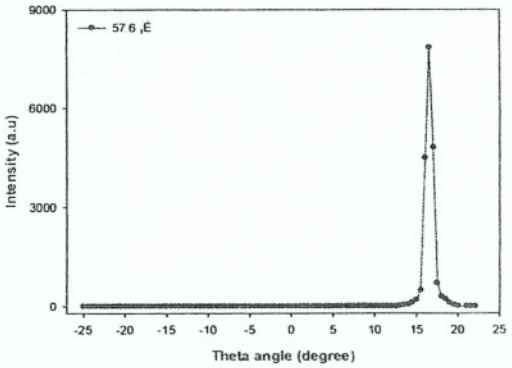

Fig. 5. Theta scan of PSCOF cell made by binary mixture of Felix-015/100:NOA65=70:30 at 57.6 ℃.

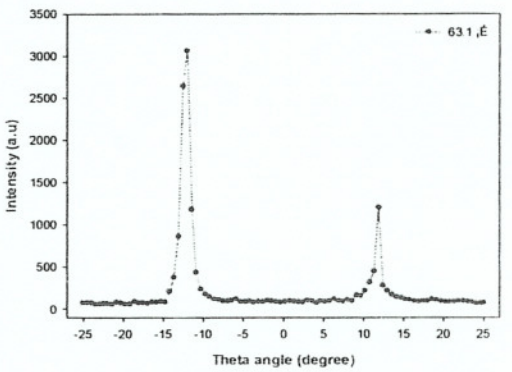

Fig. 6. Theta scan of SSFLC cell made by pure Felix-016/100 FLC at 63.1 ℃. The peak positions are symmetric but the intensity of the peaks is not symmetric.

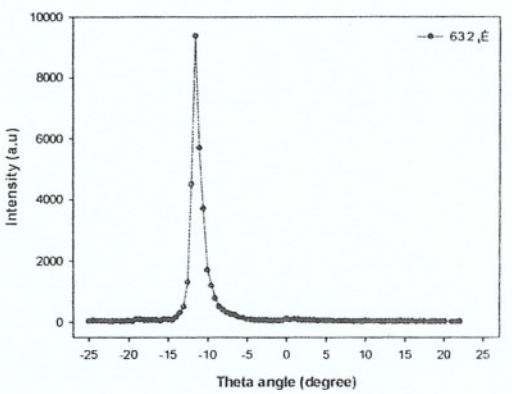

Fig. 7. Theta scan of PSCOF cell made by mixture of Felix-016/100:NOA65=70:30 at 63.2 ℃.

4. Conclusion

We discussed the layer structure obtained by x-ray measurements in PSCOF cell on the ratio of concentration between liquid crystal and pre-polymer. We found that the best ratio of concentration between liquid crystal and pre-polymer is 70wt% of liquid crystal and 30wt% of pre-polymer from electro-optical characteristics.

The layer structure in PSCOF cell is tilt-bookshelf structure in both samples which gives information of free zig-zag defect. The angle of tilt-bookshelf layer structure is almost same of tilt angle of ferroelectric liquid crystal in Sm C* phase for compensating layer buckling. We are now in progress to analyze the layer structure on various ratio of concentration between FLC and pre-polymer. And, we will find the mechanism why PSCOF technology can have results of free zig-zag defect whose technology can become a solution for future AV application.

5. Acknowledgement

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6. References

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