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Short Note

Investigation of Time-Resolved X-Ray Diffraction Analysis in Smectic *A* Liquid Crystal Cells

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Time-resolved X-ray diffraction analysis of smectic *A* liquid crystal cells is investigated. Frequency and duty ratio dependence of smectic *A* layer motions is measured. From the experimental results, possible mechanisms of layer motions are discussed.

KEYWORDS: liquid crystal, smectic *A*, X-ray diffraction, time-resolved analysis

The investigation of liquid crystal (LC) applications, especially in display devices, has progressed significantly due to the simplicity of electro-optic conversion. One of LC subjects is the high-speed response. The electroclinic effect[1] of smectic *A* (S_A) phase is attractive due to high-speed and linear response. In 1987, Garoff and Meyer discovered the electroclinic effect.[1] As a results in evaluation of molecular motion, optical modulation characteristics,[2] giant electroclinic effect[3] and applications of optical modulation at video rate[4,5] were reported. As a results in the evaluation of layer motions, the layer structure of S_A LC[6] and the layer deformations under electric field application[7–11] were also investigated, in a similar manner as evaluation of a ferroelectric liquid crystal (FLC) cell by Rieker *et al.*[12] As regards layer deformation analysis, the reversible layer deformation along the cell thickness direction, the correspondence of molecular tilt angle and layer tilt angle, and the possibility of the influence of the layer motion and two-component electro-optical responses have been reported by Johno *et al.*[7] Shao *et al.*,[8] Skarp *et al.*,[9] Crawford *et al.*,[10] and Rappaport *et al.*[11] also reported the winding of layer to the layer normal direction. In this study, we investigate the layer motion of smectic *A* LC cells by time-resolved X-ray diffraction (TRXRD) analysis.

The LC used was TM-C108 (Chisso, Cryst(20.8°C)S_A (73.8°C)Iso). The alignment layer of LC molecules consisted of polyacrylonitrile (PAN). The pretilt angle of a typical nematic material in the cell with PAN alignment layer was 2°. The rubbing direction was antiparallel and cell thickness was 2 μm. The LC cell temperature was 27°C. The X-ray diffraction system used was RINT-1100 (Rigaku, conditions: 40 kV, 30 mA). The thickness of the glass substrate was 60 μm.

Figure 1 shows the measurement system. A window pulse for signal selection is generated from the applied pulse voltage with delay time t_D. Time-resolved analysis can be carried out by synchronizing the output selection of the scintillation counter (S.C.) with the window pulse.

Before we investigated the dynamic layer motion, X-ray diffraction measurement under DC voltage application was carried out in a manner similar to that used by Rieker *et al.*[12] and Johno *et al.*[7] Figure 2 shows the X-ray diffraction intensity versus cell rotation angle characteristics with applied DC voltage varied from 0 V to 8 V. Bragg angle $2\theta_B$ was fixed at 3.4°. The cell rotation angle was varied from 75° to 105°. Without applied voltage, a single peak corresponding to the bookshelf structure was observed. Under an applied voltage of above 4 V, two additional peaks corresponding to the chevron structure appeared. The layer tilt angle δ ($= \alpha - \theta_B$) and the peak intensities corresponding to the chevron structure gradually increased with applied DC voltage. Under an applied voltage of 8 V, the layer tilt angle was about 6.5°. After DC voltage application was terminated, the layer structure reverted to the initial bookshelf structure.

The TRXRD analysis is carried out to clarify the behavior of the layer motion with time. The cell rotation angle was varied from 80° to 100°. The scan speed was 2°/min at frequencies of 1, 10 and 100 Hz and 0.2°/min at 0.1 Hz. The sampling period was 0.05°. The number of sampling integrals was 150, 15, 1.5 and 1.5 times at 100, 10, 1 and 0.1 Hz, respectively. Figure 3 shows the results of TRXRD analysis under the pulse waveform with

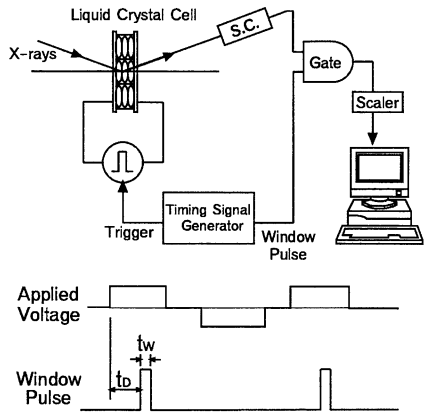

Fig. 1. Time resolved X-ray diffraction system under study.

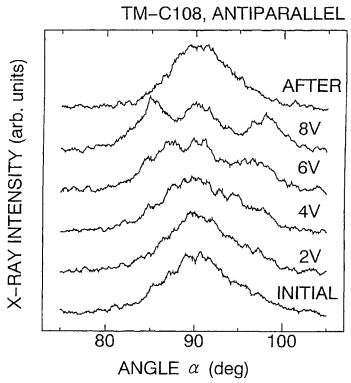

Fig. 2. X-ray diffraction intensity versus cell rotation angle with applied DC voltage, as parameter.

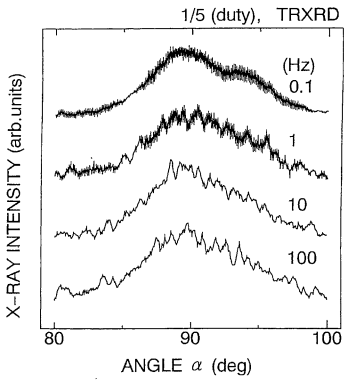

Fig. 3. TRXRD results with frequency, as parameter.

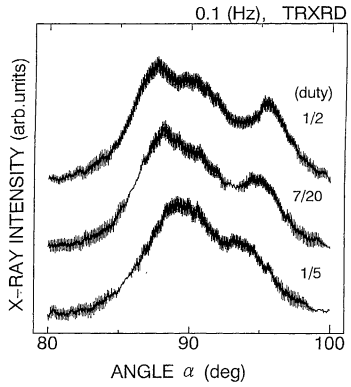

Fig. 4. TRXRD results with duty ratio, as parameter.

1/5 duty ratio, amplitude of ±8 V, delay time $t_D = 0$ and same window pulse width t_W as that of the positive polarity pulse. The frequencies were 0.1, 1, 10 and 100 Hz. The peak position and its intensity were almost the same when the frequency was varied from 1 Hz to 100 Hz, but was different in the case of frequency as 0.1 Hz. At 0.1 and 1 Hz, the small fluctuation of the intensity was related to the asynchronous sampling variation with the stage rotation. Figure 4 shows the results of TRXRD analysis with duty ratio as a parameter. The amplitude of the bipolar pulse was 8 V. With increasing the duty ratio, two peaks corresponding to the chevron structure appeared and the layer tilt angle increased. TRXRD analysis was carried out under another condition: the cell rotation angle was varied from 80° to 100°. The duty ratio and amplitude of the pulse waveform were the same as the above experiment. The frequency was 100 Hz. The duty ratio of the window pulse width was 1/100. The delay time t_D was varied from $-200\,\mu$s to 1.2 ms. However, these curves of X-ray intensities that were obtained by varying delay time were almost the same.

X-ray measurement of another LC cell was also carried out under rectangular pulse voltage of 10 V, 1/2 duty ratio and frequency ranging from 100 Hz to 1 MHz. The initial layer without applied voltage exhibited the bookshelf structure. Chevron layer deformation was observed at all frequencies investigated.

The mechanisms of layer motion from X-ray diffraction analysis are discussed. Possible mechanisms of layer motion are as follows: (1) reversible layer switching between bookshelf structure and chevron structure takes place, and (2) layer structure switching is related to the duty ratio of the applied voltage but is not related to the time scale. From Fig. 3, no obvious structural change of layers was observed when frequency was varied from 1 Hz to 100 Hz; structural change of layers was observed only at 0.1 Hz. With increasing duty ratio, as shown in Fig. 4, marked layer structure deformation was observed. The layer reverted to the initial bookshelf structure without voltage application. From Figs. 3 and 4, in case the structural change in Fig. 3 was valid, one of the time constants of layer motion was as slow as around several seconds. This time constant was of the same order as that from the estimation of indirect layer reorientation in ferroelectric liquid crystal.[13] As shown in Fig. 3, from 100 to 1 Hz, no layer deformation was observed at 1/5 duty ratio. Under rectangular pulse application with 1/2 duty ratio, however, large layer deformation was observed. Therefore, the mechanism of duty-ratio-dependent layer deformation is also supported.

Direct observation of layer switching in smectic *A* LC cells was conducted by time-resolved X-ray diffraction analysis. TRXRD analysis in which delay time and window pulse width are varied and the correspondence of layer switching to the electro-optical measurement are our future subjects. In this study, a series of experiments was carried out under the same conditions, especially in the sampling integral time. To obtain a valid explanation of layer motion, accurate evaluation of the time constant of layer motion under a high-intensity X-ray source, modeling of layer motion, and study of molecular and layer reorientation are necessary.

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