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## Dynamic Layer Response under Electric Field in Antiferroelectric Liquid Crystal Cells Measured by Synchrotron Microbeam Time Resolved X-Ray Diffraction

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# Dynamic Layer Response under Electric Field in Antiferroelectric Liquid Crystal Cells Measured by Synchrotron Microbeam Time Resolved X-Ray Diffraction

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Direct observation of the local layer response of an antiferroelectric liquid crystal to the step form electric field has been carried out by a time resolved synchrotron X-ray micro diffraction measurement. When an electric field was changed from high voltage to 0V, corresponding to the ferroelectric to antiferroelectric phase transition, the local layer transformed from the bookshelf to the quasi-bookshelf structure within 0.3 ms. The horizontal chevron structure was found in both the phases, though the decrease in the horizontal chevron angle was observed during a period of 0.2 ms after turning off the electric field. In the antiferroelectric to ferroelectric phase transition process (from 0V to high voltage), the layer structure transformed to the bookshelf within 0.04 ms.

**Keywords:** X-ray microbeam; time resolved measurement; X-ray diffraction; antiferroelectric liquid crystal; phase transition; local layer structure

## INTRODUCTION

Since the electric field induced phase transition between the

antiferroelectric and ferroelectric phases was discovered in antiferroelectric liquid crystal (AFLC) cells[1], the dynamic responses of the director and the layer structure upon applying an electric field have been of great interest both for display device applications and for basic physics. Though many optical and electrical measurements have been made to clarify the phase transition mechanism[2,3], detailed and direct information about the local layer structure and its response to the electric field has been limited. The characterization using X-ray small angle diffraction is desirable for the study of the local layer structure. Since the variation of texture in the smectic phase is expected to be influenced by the local layer structure, the spatial resolution in addition to the time resolution is necessary to investigate the layer response to the electric field. Recently, synchrotron X-ray microbeam diffraction has been successfully applied to the analysis of the smectic texture and the layer response[4-9].

In a separate paper[10], the local layer response of AFLC to the triangular electric field has been reported, and the horizontal chevron structure has been confirmed during the field induced transition. In this study, the transient local layer response between the ferroelectric (FE) and antiferroelectric (AFE) phases under a step form electric field was measured using a time resolved X-ray microbeam diffraction system.

## **EXPERIMENTAL**

The experiments were carried out at the Photon Factory on beam-line 4A. The X-ray energy was 8 keV and the X-ray beam size at the sample was about  $3 \times 4 \mu\text{m}^2$ [11]. A position sensitive proportional counter (PSPC) was used for the X-ray detector. The diffraction geometry is schematically shown in Figure 1. The  $\omega$ -angle corresponds to a layer tilt angle from the cell surface while the  $\chi$ -angle is a layer tilt angle with respect to the rubbing direction in the cell surface plane. X-ray diffraction data were collected synchronized with an applied electric field, which was a step form with 50Hz and 100Hz,  $\pm 50\text{V}$ .

The sample was TFMHPOBC (AFLC) and sandwiched between ITO-coated glass plates rubbed one-side after coating a polyimide alignment film. The cell gap was about  $5 \mu\text{m}$ . The sample was homogeneously aligned by an electric field treatment in the  $\text{SmC}_A^*$  phase. The aligned sample was once heated to the  $\text{SmA}$  phase and then cooled down to  $T_c - 10^\circ\text{C}$  to perform the experiments, where  $T_c$  is the

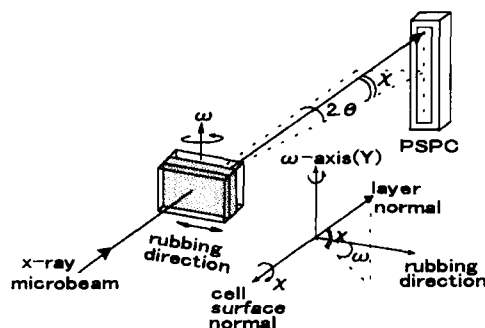


FIGURE 1 Experimental setup. The  $\omega$ -scan profile (rocking curve) was measured by the sample rotation around the vertical axis. The  $\chi$ -intensity profile was collected by a position sensitive proportional counter (PSPC) at fixed  $\omega$ -angles.

phase transition temperature from SmA to SmC<sub>A</sub>\* (109°C[12]) and, in practice, was confirmed by the in-situ optical microscope observation.

## RESULTS AND DISCUSSION

In the initial state of the SmC<sub>A</sub>\* phase, the sample had the stripe defect texture running parallel to the rubbing direction. The irreversible layer transition from the initial state to the high electric field state is briefly summarized[10]. In the initial state, the  $\omega$ -profile shows triple peaks (Figure 2(a)) and is explained by a combination of vertical chevron (v-chevron) and horizontal chevron (h-chevron) structures. The v-chevron angle ( $\delta$ ), defined as half the angular difference between a pair of peaks, was 15 deg. The h-chevron angle ( $\alpha$ ), determined from the  $\chi$ -profile at  $\omega=0$  deg, was 20 deg. When the amplitude of the step form electric field (50Hz) was increased, the layer structure changed irreversibly to the vertical bookshelf (v-bookshelf) at a high electric field ( $\pm 50V$ ) (Figure 2(b)). After the irreversible transformation, the layer structure shows the reversible transition between the v-bookshelf and the quasi-bookshelf structure under the application of high and low fields (Figures 2(b) and 2(c)), respectively. At the high field, the  $\omega$ -profile indicates the small v-chevron peaks with a reduced angle of  $\delta=3$  deg in addition to the bookshelf peak at  $\omega=0$  deg. The stable h-chevron with a

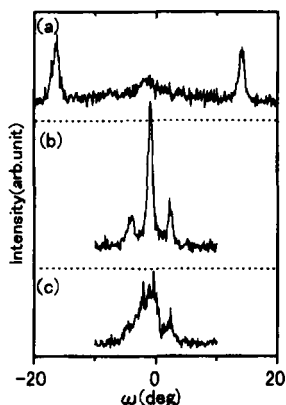


FIGURE 2 A series of time integrated  $\omega$ -scan profiles corresponding to the initial state (a), during the field application (step form, 50Hz,  $\pm 50$ V) (b) and after turning off the field (c).

reduced h-chevron angle ( $\alpha$ ) of 5 deg was observed even under a low field. The small  $\delta$  and  $\alpha$  after the irreversible transformation mean the reorientation of molecules and the change in the layer structure.

A spatial distribution of the time integrated  $\omega$  and  $\chi$ -profiles was measured under the step form electric field application by changing the analysing position with 3- $\mu\text{m}$  step perpendicular to the stripe texture (Figure 3). A series of  $\omega$ -profiles (Figure 3(a)) shows the v-bookshelf structure with the partial v-chevron independent of the analysing position. The  $\chi$ -profiles (Figure 3(b)) clearly show the positional dependence and are related to the stripe texture. These results are consistent with those obtained under a triangular electric field except for fine structures[10].

The time resolved X-ray microbeam diffraction profiles were measured for the step form electric field (50 and 100 Hz,  $\pm 50$ V). The changes in the  $\omega$  and  $\chi$  profiles were obtained for the FE (50V) to AFE (0V) phase transition as a function of time with a 0.1 ms time resolution. The  $\omega$ -profiles (Figure 4(a)) indicate that the v-bookshelf peak becomes slightly broad (5.0–5.2 ms) and then the weak v-chevron peaks gradually increase in addition to the bookshelf peak (5.3 ms–).

The X-ray intensities in the  $\omega$ -profile at various analyzing positions are summarised as a function of time in Figure 5. Though the response profile depends on the position, the 0.3 ms transient time on an average was confirmed. The transient time can be also characterized as the appearance of the weak v-chevron (at 5.3 ms). The positional dependence may be caused by the spatial layer modulation of the stripe texture in addition to the domain nucleation process.

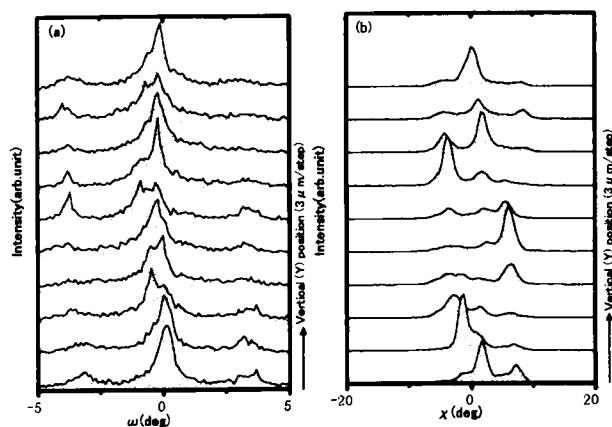


FIGURE 3 A series of time integrated  $\omega$  (a) and  $\chi$  (b) profiles during the application of a step form electric field (50Hz,  $\pm 50$ V) as a function of analysing position across the stripe texture measured by  $3 \mu\text{m}/\text{step}$ .  $\chi$ -profiles were collected for 100 s at  $\omega$ -peak positions in each step.

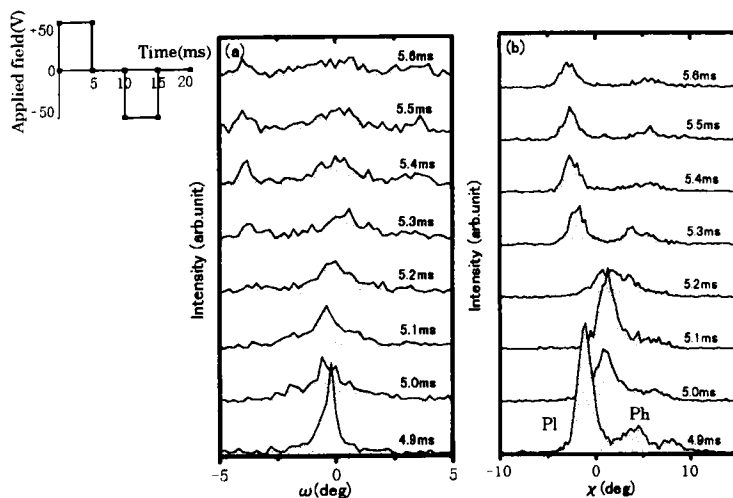


FIGURE 4 Time resolved X-ray microbeam diffraction intensity profiles for  $\omega$  (a) and  $\chi$  (b) directions for the FE (50V) to AFE (0V) phase transition process.  $\chi$ -profiles were obtained at  $\omega = -0.3 \text{ deg}$ . Time resolution was 0.1 ms.

The  $\chi$  distribution (Figure 4(b)) was measured at the bookshelf peak position ( $\omega = -0.3$  deg). Just after turning off the field (5.0–5.2 ms), the h-chevron angle ( $\alpha$ ) decreases as a function of time. As shown in Figure 4(b), the horizontal bookshelf ( $\alpha \cong 0$  deg) was realized at 5.2 ms. The horizontal layer structure at 5.2 ms, however, depends on the analyzing position. The h-chevron with the small chevron angle still remained in other positions. After 5.3 ms, the layer structure forms the h-chevron again. The transient time from the high field to the low field h-chevron in the  $\chi$ -direction is also about 0.3 ms.

Both the  $\omega$  and  $\chi$  profiles indicate that the layer structure transforms continuously from 5.0 ms to 5.2 ms, and changes discontinuously at 5.3 ms. From these observations, the transient layer structure (5.0–5.2 ms) seems to be realized in the FE phase and the quasi-bookshelf with the h-chevron after 5.3 ms corresponds to the layer structure in the AFE phase. Time resolved diffraction study for a ferroelectric liquid crystals would reveal this transient process in the FE phase more clearly.

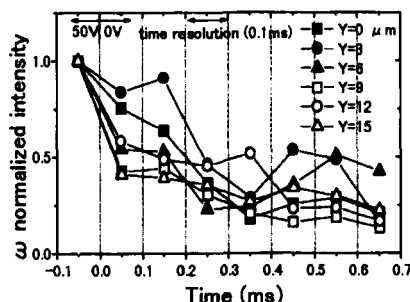


FIGURE 5 The X-ray intensities in the  $\omega$ -profile at  $\omega = -0.3$  deg as a function of time. The analyzing position was changed across the stripe texture by  $3 \mu\text{m}/\text{step}$ .

For the AFE (0V) to FE (-50V) phase transition, the layer response was much faster than the reverse process. The time resolved  $\omega$ -profiles with a 0.02 ms time resolution (Figure 6(a)) show the quasi-bookshelf to the bookshelf transformation. The increase of the horizontal chevron structure is observed in the time resolved  $\chi$ -profiles (Figure 6(b)). The transformation proceeds within 0.04 ms. This result



indicates that the layer is stretched quickly by the strong electric field. The  $\chi$ -distribution (Figure 6(b)) shows no transient structure within the present time resolution.

The transient time obtained in Figures 4-6 seems to correspond to the response time of the slow process in an optical response[3], though the sample and experimental conditions are different. Since the slow optical response is caused by the movement of domain boundaries, the transient time of the layer structure depends on the position. The microbeam measurement over large area is expected to clarify whether the present transient response is related with the optical response.

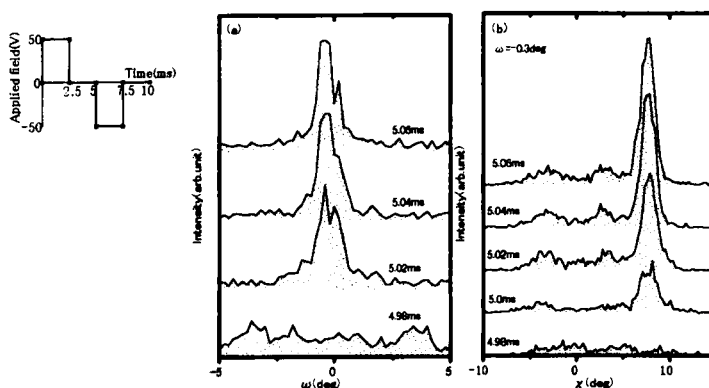


FIGURE 6 Time resolved X-ray microbeam diffraction intensity profiles for  $\omega$  (a) and  $\chi$  (b) directions for the AFE (0V) to FE (-50V) phase transition process.  $\chi$ -profiles were obtained at  $\omega = -0.3$  deg. Time resolution was 0.02 ms. Note that  $\omega$ -profile at 5.0 ms is missing.

## CONCLUSION

The transient local layer deformation process related to the field induced AFE/FE phase transition was observed, for the first time, in real time using a time resolved synchrotron X-ray microbeam diffraction.

For the FE to AFE transition, the layer structure changed from the v-bookshelf to the quasi-bookshelf within 0.3 ms. The h-chevron was stable in both states, though the h-chevron angle was decreasing till

0.2 ms after turning off the applied field. For the AFE to FE transition, the reverse layer deformation occurred within 0.04 ms. To clarify the transient local layer response, time resolved X-ray studies for the ferroelectric liquid crystal are now underway.

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