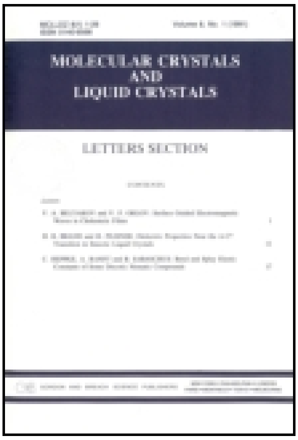


Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl20

# Pretilt Preference of Liquid Crystals on Ion-Beam-Treated Polyimide Layer Surfaces

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Published online: 16 Dec 2013.

*Mol. Cryst. Liq. Cryst.*, Vol. 583: pp. 104–108, 2013

ISSN: 1542-1406 print/1563-5287 online
DOI: 10.1080/15421406.2013.844311



# Pretilt Preference of Liquid Crystals on Ion-Beam-Treated Polyimide Layer Surfaces

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*The pre-tilt direction of a liquid crystal (LC) on a polyimide (PI) surfaces obtained by using a contact angle method is systematically examined. We found from scanning probe microscopy (damping force microscopy and friction force microscopy) data that ion-beam-treated PI surfaces have an asymmetrical morphology which determines the preferred direction of the LC. Azimuthally various contact angles on an ion-beam-treated PI surface are connected deeply with modified surface morphologies. As a result, the effect of LC pre-tilt direction on ion-beam-treated PI is to lower the contact angle direction.*

**Keywords** liquid crystal; polyimide; ion beam; scanning probe microscopy; contact angle; pre-tilt direction; roughness inclination

## Introduction

Various techniques have been developed for liquid crystal (LC) alignment such as the rubbing method,[1] ion beam alignment,[2–5] photo alignment,[6] obliquely evaporation of an inorganic film,[7] atomic force microscopy (AFM),[8] and lithography of polymers.[9] Rubbed-polyimide (PI) is still being used widely as an LC alignment in mass production of LC displays. The ion beam alignment was introduced recently to overcome the drawbacks of rubbing, such as static charges, creation of debris, and so on. However, there are still unresolved issues associated with the mechanism of LC alignment. Some attempts at describing the mechanism of the pre-tilt direction (PD) connected with an LC alignment layer have been made, based on experimental results. Stöhr *et al*. reported on PD of LC

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effects on an alignment surfaces related to the orientational bonding order in rubbed, ion-beam-treated PI, and ion-beam-treated diamond-like-carbon films, by using the near-edge x-ray absorption fine structure measurements analyses.[4]

However, we demonstrate that fine surface morphologies determine the PD of an LC, by using a contact angle method observed in ion-beam-treated PI. Here, using DFM (damping force microscopy) and FFM (friction force microscopy) equipment, we also investigated the relationship between the modified surface morphology on an ion-beam-treated PI and the contact angle of D. I. water on layer surfaces.

## Experimental

LC cells with a cell gap of 3.8 $\mu$m were fabricated by using the ion beam-based process, and a positive LC (Merck MLC-0223) was injected into the cells. Indium-tin-oxide-coated glass substrates were spin-coated with PI (RN-1702, Nissan Chemical Co.), prebaked at 80 °C for 30 min, and cured at 230 °C for 1 h. The PI surfaces were bombarded by an argon ion beam. A cold hollow cathode type ion source was used to yield the ion beam. The ion beam energy, exposure time, incident angle ($\theta$ =85 deg.) and ion beam flux density of the PI surfaces were exposed 60 eV, 1∼30 s, 85°, and $2.08\times10^{13}$ $Ar^{+}/s\cdot cm^{2}$, respectively. The x-z plane projection of the ion beam was along the -*x* axis of our coordinate system, as shown in Figure 1.

The surface morphologies of the alignment layer were determined by DFM and FFM equipment (Seiko SPA 400 instrumentation). The wetting properties of the surfaces were determined by the measurement of the static contact angle method. The azimuthal contact angles (error bar $\leq$ 0.05 deg) were measured by increasing and then decreasing the volume of a drop of D. I. water deposited on PI surfaces. Under the PI layer, the rotation stage was controlled with a resolution 0.01 degree.

## Results and Discussion

In order to search the PD of an LC on PI surfaces, we measured the azimuthal contact angle on those surfaces. Without exposure of PI surfaces to the ion beam, the azimuthal contact angle was kept typically at about 96.4° irrespective of the azimuth direction. We have found that LC molecules can be aligned vertically on PI surfaces when the exposure time is smaller than 5 s, on the other hand, they can be aligned homogeneously when it is larger than 6 s. We found that the PD of an LC on an ion-beam-treated PI surfaces takes the $\delta$ direction

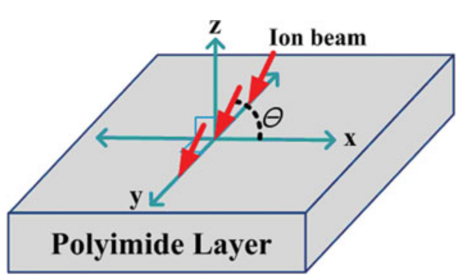


**Figure 1.** Experimental geometry of an ion-beam-treated PI (ion beam direction: $\theta = 85°$ on the x-z plane).

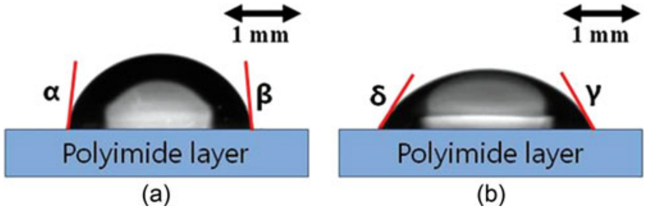


**Figure 2.** The images of (a) y axis ($\alpha$, $\beta$) and (b) x axis ($\delta$, $\gamma$) azimuthal contact angle on PI surfaces irradiated through ion beam.

using the crystal rotation method which is a simple and precise technique to determine the pre-tilt angle of a LC cell.[10] As shown in Fig. 2, with an ion beam exposure time 1 s on PI surfaces, we found that various values of azimuthal contact angle ($\alpha$: 81.42°, $\beta$: 81.42°, $\gamma$: 76.43°, and $\delta$: 69.61°) appeared on PI surfaces. Contact angles $\alpha$ and $\beta$ perpendicular to the ion beam exposure direction have symmetry with respect to the exposure direction, whilst $\gamma$ and $\delta$ with ion beam exposure direction are asymmetrical. The contact angle $\gamma$ is higher than the contact angle $\delta$. We measured the azimuthal contact angle on an ion-beam-treated PI surfaces as a function of exposure time (Table I). We found that LCs can be aligned vertically when a high contact angle (82∼78°) is observed at PI surfaces (exposure time: 1∼5 sec); the low contact angle (44∼41°) results in a homogeneous alignment (exposure time: 6∼30 sec). Of special interest is the fact that, with an exposure time of 3 sec and homeotropic LC alignment, the azimuthal contact angles are as follows ($\alpha$: 78.55°, $\beta$: 78.53°, $\gamma$: 71.92°, and $\delta$: 65.39°). On the other hand, for an exposure time of 6 sec with homogeneous LC alignment, the azimuthal contact angles are as follows ($\alpha$: 43.93°, $\beta$: 43.89°, $\gamma$: 39.45°, and $\delta$: 36.21°). Consequently, we can know that the pre-tilt angle of the LC stands facing the low azimuthal contact angle direction.

In order to obtain a better understanding of the azimuthal contact angles on PI alignment surfaces, DFM and FFM images of PI surfaces were used. Figure 3 shows the surface morphologies of without and with an ion-beam-treated PI obtained by DFM, respectively. There are no special direction surfaces morphologies of the PI before the treated ion beam. Consequently, it generates nearly the same azimuthal contact angles for all azimuthal directions on PI surface. On the other hand, after ion beam exposure, we found partly anisotropy along the exposure direction on PI surface. Eventually, partly anisotropy surface structure results in the formation of anisotropic contact angles and describes the symmetry of the azimuthal contact angle ($\alpha$, $\beta$) on PI surfaces irradiated through an ion beam.[11] Also, we investigated the cause of the asymmetry of the contact angles $\gamma$ and $\delta$ on PI surfaces irradiated through an ion beam incident angle. Figure 4 shows the FFM images obtained by

**Table 1.** The various azimuthal contact angles on PI surfaces as a function of exposure time.

| Exposure time (sec) | $\alpha$ | $\beta$ | $\gamma$ | $\delta$ |
|---|---|---|---|---|
| 1 | 81.42° | 81.42° | 76.43° | 69.61° |
| 3 | 78.55° | 78.53° | 71.92° | 65.39° |
| 6 | 43.93° | 43.89° | 39.45° | 36.21° |
| 30 | 41.47° | 41.45° | 37.79° | 35.72° |

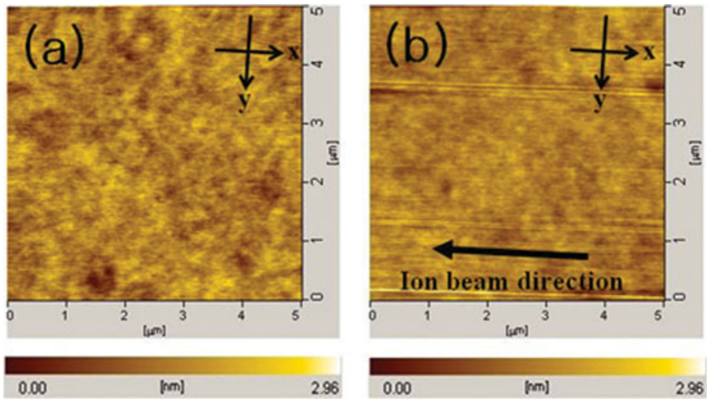


**Figure 3.** The AFM images of PI surfaces (a) before an ion beam exposure and (b) after an ion beam exposure time 1 s.

scanning in the -x direction and +x directions on PI surfaces. What is generally described as a FFM is simply a DFM which is used to measure both the vertical (normal) force as well as the lateral (friction) force in contact mode imaging. Beam deflection systems with quadrant photodiode detectors are employed for these kinds of experiments. Friction measurements are performed by imaging in contact mode, usually with constant force, and recording the lateral deflection of the cantilever which corresponds to the friction image. A topographic image is often recorded simultaneously, by measuring the feedback signal needed to maintain constant force, so that the topographical features of the sample can be compared with the tribological features appearing in the friction image.[11] The images show that the PI surface roughness inclination in the -x direction is larger than those in the +x direction. These results explain the reason why the contact angle value is higher in the -x direction than in the +x direction on PI surfaces. Namely, In addition, we measured the roughness inclination r.m.s. of the FFM data on PI surfaces as the function of exposure time as shown in Figure 5. Before the ion beam exposure, the roughness inclination was nearly identical for the two directions. However, when the exposure time was more than

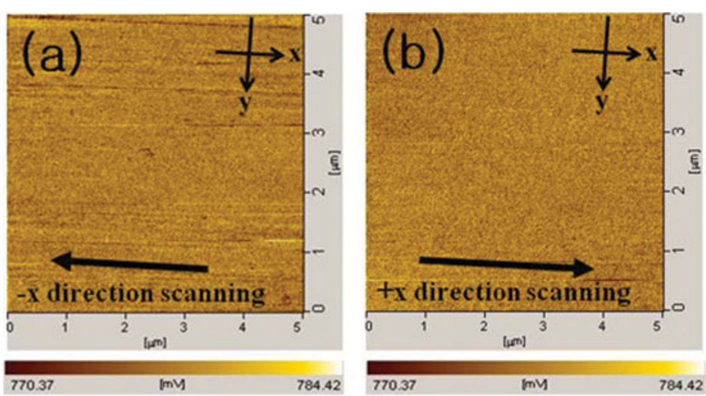


**Figure 4.** The FFM images of scanning in (a) -x direction and (b) +x direction on ion beam treated PI surfaces.

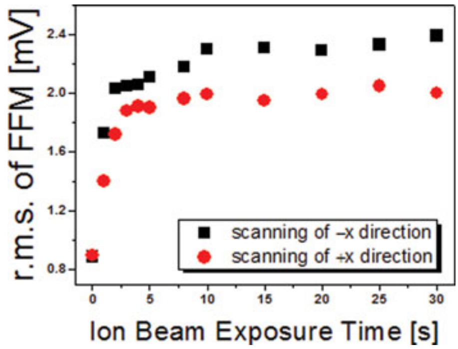


**Figure 5.** The r.m.s. data of FFM for PI surfaces as a function of exposure time.

1 s, it is evident that the roughness inclination of -x direction is larger than it is in the +x direction. The fine difference in the surface roughness inclination in the directions parallel and anti-parallel to the ion beam exposure direction generates the pre-tilt of the LC director, which aligns with the direction of low roughness inclination. This result suggests that the LC alignment mechanism is primarily induced by the surface roughness inclination.

## Conclusions

We examined systematically the PD of a LC on an ion-beam-treated PI surfaces by using contact angle, DFM, and FFM data. Ion-beam-treated PI surfaces have asymmetry morphologies which determine the preferred PD of LC and various azimuthal contact angles on ion-beam-treated PI surface are derived from the modified surface morphologies inclination. As a result, the pre-tilt of the LC director aligns to the direction of low roughness inclination and low contact angle.

## Acknowledgment

This work was supported by a grant from Kyung Hee University in 2012 (KHU-20120184).

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