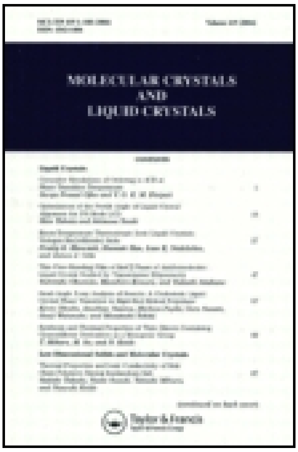

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Investigations on Langmuir-Blodgett Films as Alignment Layers for Liquid Crystals

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Investigations on Langmuir-Blodgett Films as Alignment Layers for Liquid Crystals

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Multilayer polyimide (PI) films were successfully fabricated using the Langmuir-Blodgett (LB) technique. These films were studied in several ways relevant to their use as liquid crystal alignment layers. (1) The influence of dipping speed and creep time on the orientational order of PI-LB films was investigated by means of birefringence measurements. (2) For comparison, we measured the pretilt angles and polar anchoring strengths of liquid crystal (LC) cells assembled with PI-LB films and rubbed PI-LB films as alignment layers. (3) The anchoring direction of the liquid crystal was found to be solely determined by the dipping direction of uppermost PI-LB layer, regardless of the dipping direction of deeper layers. (4) Combining results of the above studies with measurements of pretransitional birefringence (above the isotropic to nematic (I → N) transition) and observations of the growth of the alignment texture just below the nematic-isotropic transition, we draw the conclusion that the range of interaction between the PI-LB film and LC molecules is quite short (~ 4.5 Å) and that the alignment mechanism is epitaxial. Therefore, anisotropic short range molecular interactions are responsible for the alignment of the first liquid crystal layer. (5) From studies of PI-LB films deposited perpendicular to the rubbing direction of underlying spin-coated PI films, we also found evidence, as expected, that the grooves induced by the rubbing process are not decisive for LC alignment on a rubbed polymer surface.

Keywords: Liquid crystals, alignment, LB films

1. INTRODUCTION

The wide application of flat displays using liquid crystals (LCDs) has stimulated extensive study of alignment.[1–3] Conventional processes for forming homogenous alignment layers include a rubbed polymer coating and oblique evaporation of SiO_x^2. Although the oblique evaporation method has the advantage of giving the liquid crystal molecules a prescribed pretilt angle, the deposition process is quite complicated and it is difficult to obtain a large uniform aligning area. The rubbing process also has several serious shortcomings.[4,5] Static electricity generated by rubbing causes flicker during operation of LCDs. Furthermore, the delicate switching elements on the surface, for example, thin film transistors, can be destroyed by the rubbing process and rubbing can introduce dust particles which can degrade the performance of LCDs. For these reasons, researchers have tried to develop alternative methods of alignment. Ordered polyimide Langmuir-Blodgett (PI-LB) films have recently been shown to have enormous promise as LCD alignment layers.[6-11] It is now clear that LB films with their well-controlled architecture, large-area capability and dust free character are contenders for the alignment layers of future LCDs. Due to this potential application in the LCD industry, we need to know the mechanism of alignment and how to control the important parameters for display, for example, anchoring strength and pretilt angle.

In this paper, we present our preliminary results on PI-LB film alignment layers. Orientational order of PI-LB films was investigated by means of the birefringence measurements on films prepared with different dipping speeds and creep times. The pretilt angle and the temperature dependence of the polar anchoring strength of liquid crystal cells assembled with PI-LB alignment films and their rubbed counterparts were also measured. We have also made the first study of the pretransitional birefringence of LC cells with PI-LB alignment films. Based on our experimental results, the LC alignment mechanism on PI-LB films is suggested. Finally, we bring into focus the advantages and problems associated with PI-LB films as alignment layers. Possible solutions for the existing problems are suggested.

2. EXPERIMENTAL

2.1 Preparation of multilayer PI-LB films

Figure 1 shows the flow chart of the PI-LB preparation. LB films were prepared in a 70 cm × 15 cm Lauda trough. Due to the non-amphiphilic nature of the PI, we first mixed poly[4,4'-oxydiphenylene] pyromellitamic acid (I) with N,N 'dimethylhexa decylamine (II) in the molar ratio of 1:2 in an organic solvent of N,N-dimethylacetamide and benzene (1:1 in a molar ratio) to form a poly[4,4'-oxydiphenylene] pyromellitamic acid salt (III). 100 μl of (III) was then spread on deionized water at 20°C. After waiting for 10 min, the monolayer was compressed at a speed of 1.8 cm/min. The value of the co-area obtained was 110 Å^2. Y-type LB films of (III) were transferred onto the indium-tin-oxide (ITO) coated patterned glass by vertical dipping at a surface pressure of 25 mN/m and dipping speed of 1 mm/min for the first round trip and 10 mm/min for subsequent dipping. Films of (III) were immersed overnight in a mixed organic solvent of acetic anhydride, pyridine and benzene (1:1:3) for imidization to obtain polyimide (IV) LB(PI-LB) films.

2.2 Measurements of the anchoring strength, pretilt angle and optical retardation

The liquid crystal cells were made with two identical ITO-patterned glass substrates coated with PI-LB films. We assembled the cells with the dipping direction of the last LB layer antiparallel to each other. The thickness (~ 50 μm) of each cell was determined by the optical interference method (± 0.5 μm) before the cells were filled with 4-cyano-4'-n-pentybiphenyl (5CB) liquid crystal by the capillary method. These cells were heated to 80°C (above the nematic-isotropic clearing point) then slowly

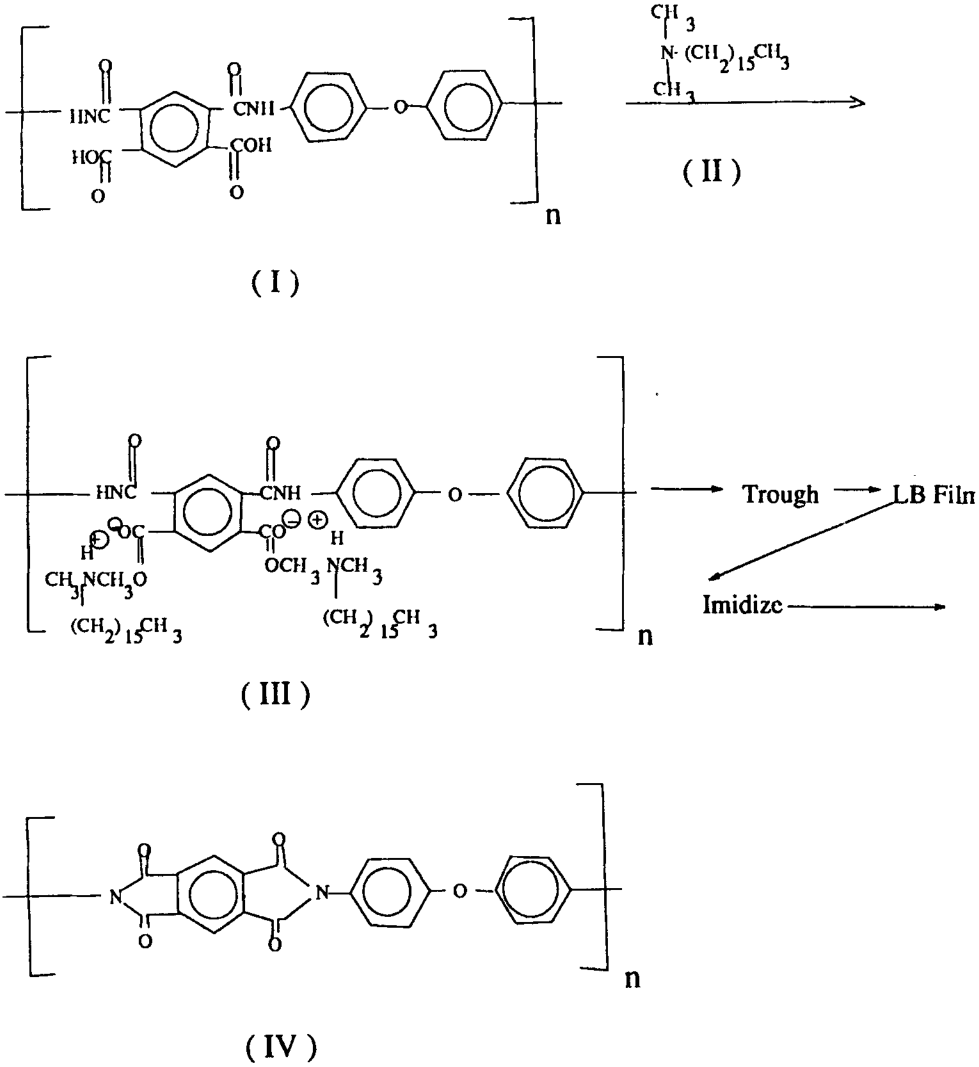

FIGURE 1 Flow chart of the multilayer PI-LB film preparation.

cooled down to room temperature to get rid of the flow alignment effect induced by filling the cell. 5CB was purchased from BDH chemicals and used without further purification.

The optical retardation of the PI-LB film was measured by ellipsometry with a resolution of 0.01°. The standard crystal rotation method[12] was used to determine the pretilt angle. To measure the polar anchoring strength, we adopted Yokoyama and VanSprang's high field method,[13,14] which requires simultaneous measurements of the voltage dependence of the birefringence and capacitance of a homogeneously aligned nematic LC cell. The schematic diagram for the optical phase retardation and capacitance measurement apparatus is shown in Figure 2.

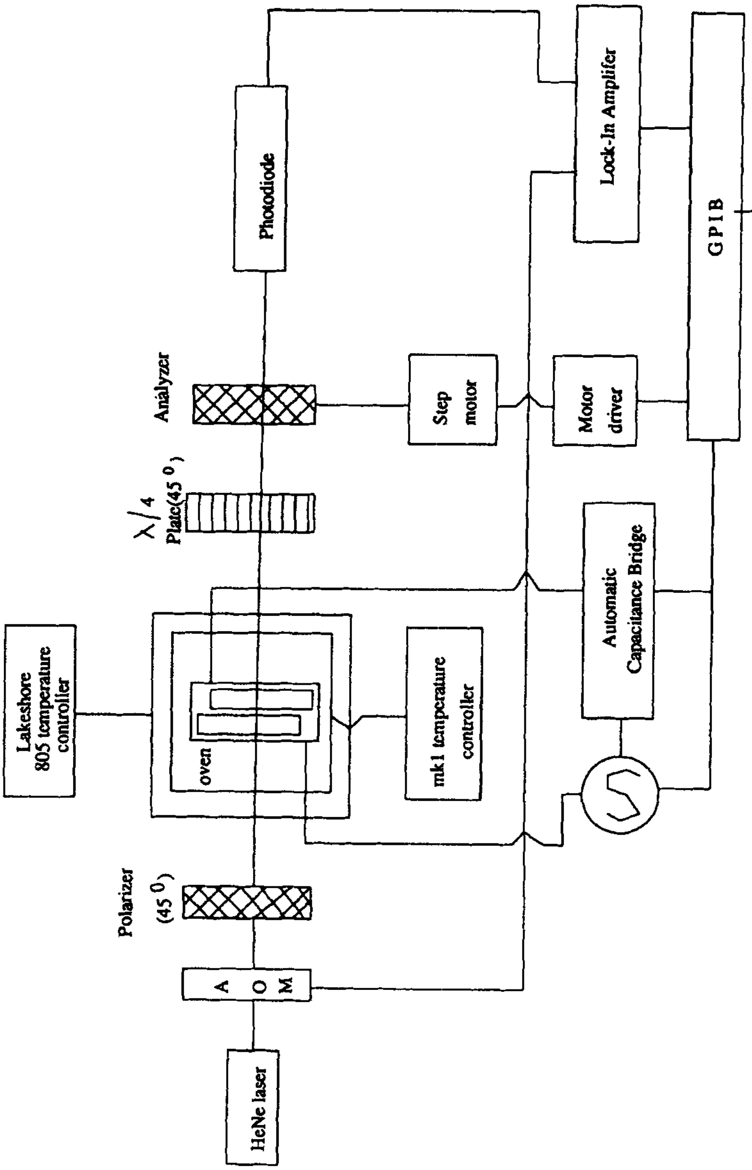

FIGURE 2 Experimental set-up for the optical phase retardation and capacitance measurements. All elements are self explanatory except the one labeled AOM which stands for Acoustic Optic Modulator.

For applied voltages, V, larger than 6 times the Freedericksz threshold voltage, V_{th}, the following relation should be accurate:[13]

$$R/R_0 = I_0/CV - 2d_e/d_0 \tag{1}$$

where the constant I_0 is related to material parameters. R_0 is equal to $2\pi d_0(n_e - n_o)/\lambda$. d_e is called the surface extrapolation length. The polar anchoring strength, W_0 is directly related to d_e. In particular, for the Rapini-Papoular surface potential, $\frac{1}{2}W_0\sin^2(\theta - \theta_e)$, we have a simple relationship $W_0 = K_1/d_e$, where K_1 is the splay elastic constant of the LC which was measured separately. C and d_0 are the capacitance and thickness of the LC cell respectively. n_0 and n_e are the ordinary and extraordinary refractive indices of the LC. Temperature was controlled to ± 5 mk over 10 h. For the measurement of the pretransitional birefringence, the temperature was swept at the rate of 0.18° C/h and the result was independent of sweep rate at this rate.

3. RESULTS AND DISCUSSIONS

At least 3 PI-LB layers were needed to achieve good uniform LC alignment on PI-LB/ITO glass. The reason may be that 3 layers are enough to screen the interaction between the LC and ITO glass surfaces, and smoothly and fully cover the surface. All LC cells made with 3 to 15 PI-LB layers gave identical, good homogenous alignment. In all cases, the LC alignment direction matched with the dipping direction of LB deposition. To check the effectiveness of alignment of the last PI-LB layer, we deposited 6 layers on glass plates in such a way that the dipping direction of the sixth layer was changed by 45° with respect to the dipping direction of the previous five layers. Polarized microscopy showed that the LC anchoring direction was solely determined by the dipping direction of the sixth PI layer, which means that the interaction responsible for LC alignment on PI-LB films is rather short ranged, i.e, less than the thickness of one PI layer (~ 4.5 Å as measured by our group.[15])

As is known, LC alignment on a PI-LB film is induced by the well ordered polymer, which aligns along the dipping direction.[16] The dipping speed is expected to be an important parameter for controlling the quality of the LB film. The influence of dipping speed and creep time on the order of PI-LB films was investigated by measuring the optical retardation. The result is shown in Figure 3. Because of the birefringence of the ITO glass itself, microscope slides were used. Assuming that the retardation $R = 2\pi d_0(n_e - n_o)S/\lambda$, where S is an appropriately defined orientational order parameter of the PI-LB film, and n_e and n_o are the extraordinary and ordinary refractive indices of the polymer respectively, it seems that in our range of dipping speed, the order of the LB film doesn't change. Furthermore, we did not see any difference in retardation of the PI-LB films for creep times of 1 hour and 2 hours.

The pretilt angles of LC cells with various numbers of LB layers were measured by the standard crystal rotation method.[12] The results are presented in Figure 4. All samples show very small or zero pretilt angle. When we applied an electric field across these cells, all of them showed reverse tilt domains under polarized microscopy, which confirmed the tilt angle measurement results. Introducing a finite pretilt angle will be very important for the application of LB films as alignment layers.

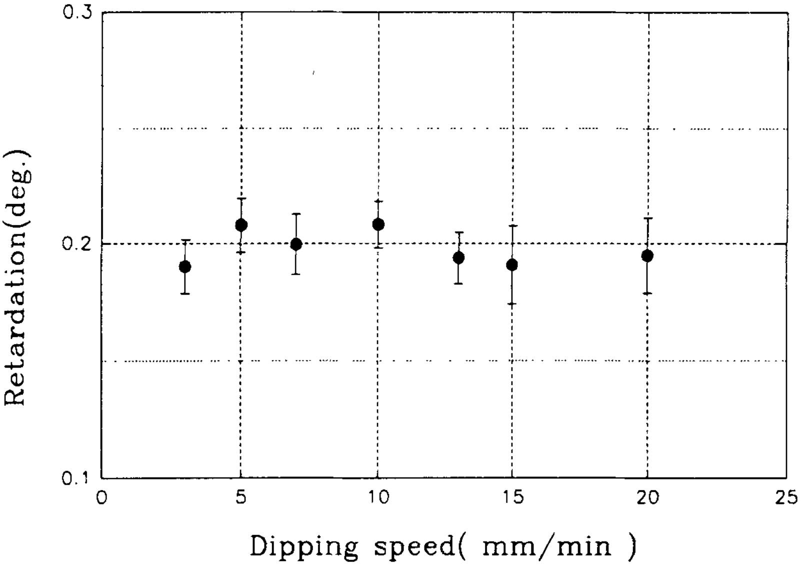

FIGURE 3 Phase retardation of PI-LB (5) film versus the dipping speed. Both sides of the microscope sheet glass have 5 layers of PI-LB films.

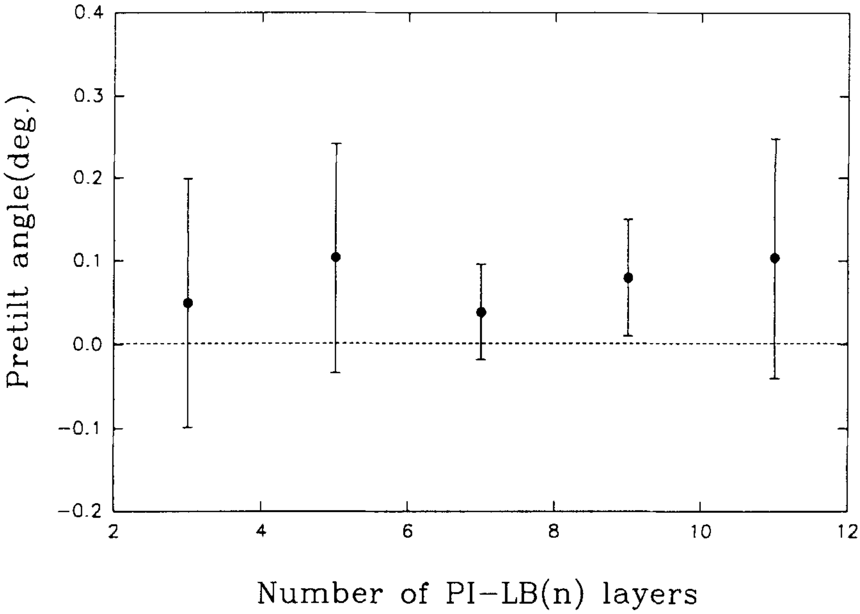

FIGURE 4 Pretilt angle versus the number n of PI-LB (n) layers.

Figure 5 shows two typical R/R_0 versus $1/CV$ curves of a liquid crystal cell with five PI-LB layers at two different temperatures. Fitting data of this type to Eq. (1), the polar anchoring strength and extrapolation length at different temperatures can be calculated and is shown in Figure 6. The polar anchoring strength of the PI-LB film is

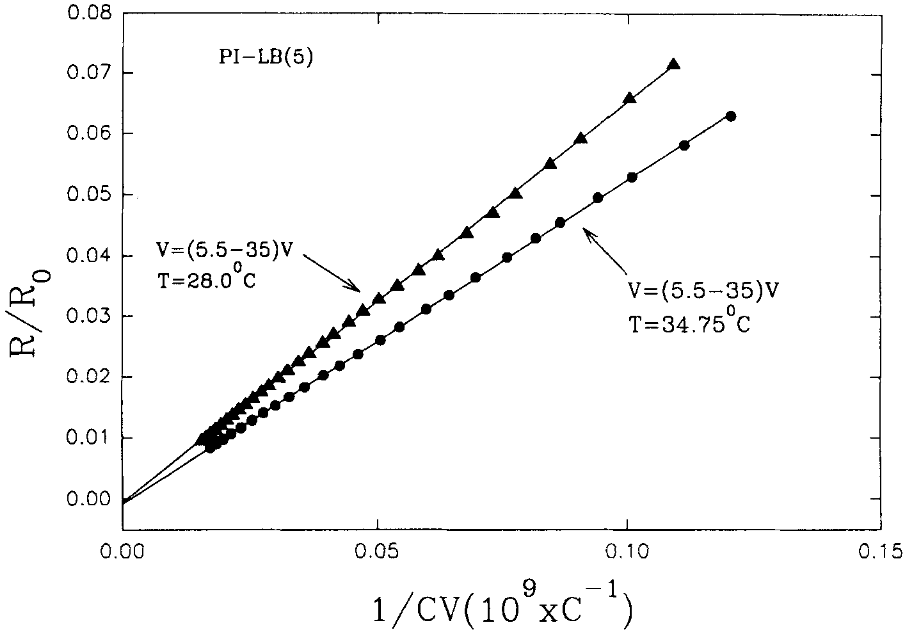

FIGURE 5 Typical R/R_0 versus $1/CV$ curves at two different temperatures.

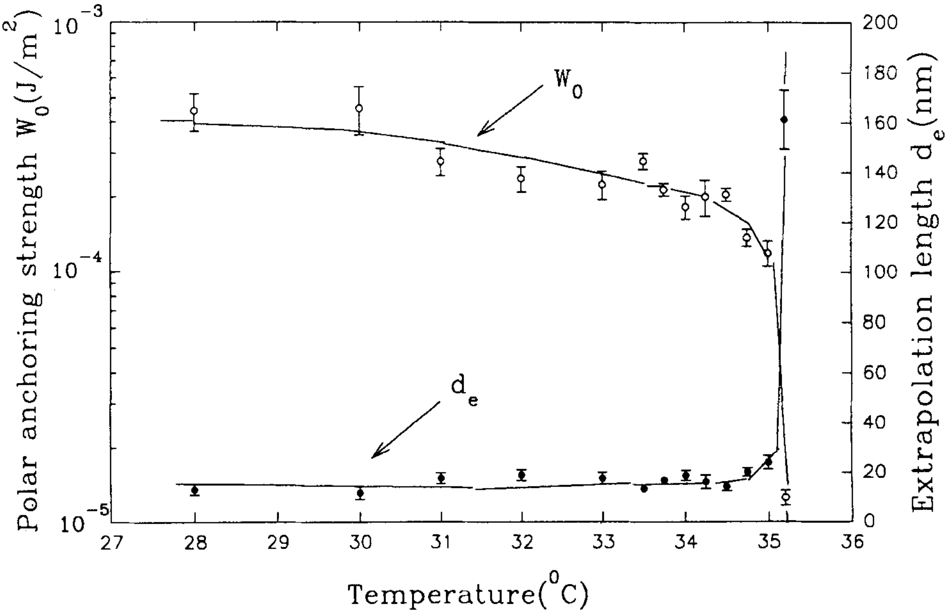

FIGURE 6 Polar anchoring energy and extrapolation length of PI-LB (5) versus temperature.

much stronger than that of SiO_x,[13,14] but somewhat weaker than that of a spin-coated rubbed polymer film.[3]

As mentioned above, PI-LB films fail to align the liquid crystal with a pretilt angle, which is a prerequisite for LCDs to avoid reverse tilt domains. One technique to introduce a pretilt angle is to manipulate the side chain density of the polyimide precursor. The number of side chains can be controlled by varying the degree of imidization. It has been claimed that in this way one can control the pretilt angle of the liquid crystal on such LB films.[18] However, so far, we don't understand how to force the side chains to flip down unidirectionally to give pretilt angles that are uniform over the cell. Furthermore, the thermal stability of the pretilt angle has not been investigated. An alternative method to get a finite pretilt angle is to rub the PI-LB film. Due to the fact that PI-LB films are very thin (about 22 Å for 5 layers), static charge accumulation should not be a problem. Figure 7 shows two crystal rotation curves of LC cells assembed with PI-LB films with and without rubbing. Clearly, the symmetry point shifted away from zero angle after rubbing. The pretilt angle obtained in this case was about 2°. For comparison, we also measured the polar anchoring strength of the rubbed PI-LB films, which is shown in Figure 8. It seems that rubbing doesn't enhance the polar anchoring strength of PI-LB films, which is not in agreement with the results of Seo *et al.*[8] We also note that the polar anchoring strength of our non-rubbed PI-LB film is almost the same as that of their rubbed one. Atomic force microscopy (AFM) images indicate that our PI-LB films are already highly ordered before rubbing.[15] Evidently rubbing does not further improve the order of the film. Based on these results, we suggest that the polymer backbones lay statistically flat on the glass surface along the dipping direction, and, therefore, they fail to align the LC with a pretilt angle. The

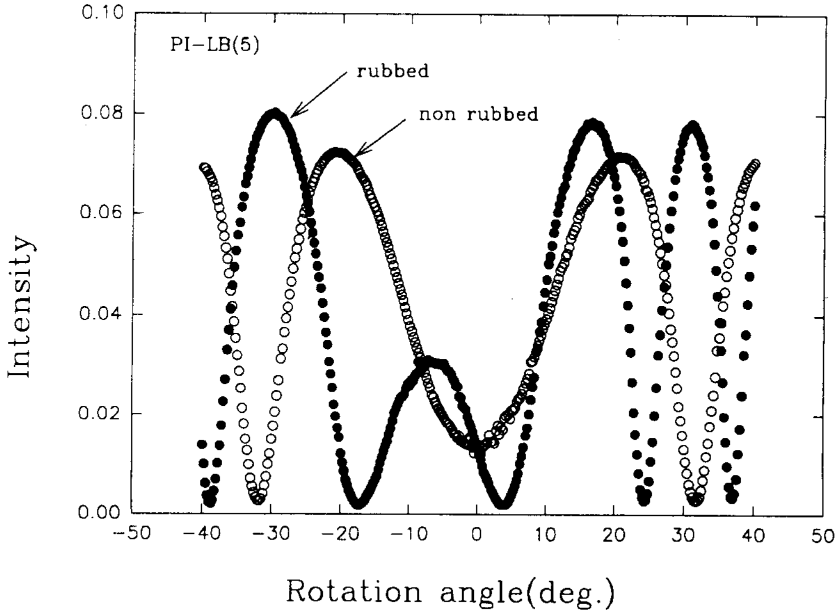

FIGURE 7 Crystal rotation curves of LC cells having PI-LB (5) films with and without rubbing.

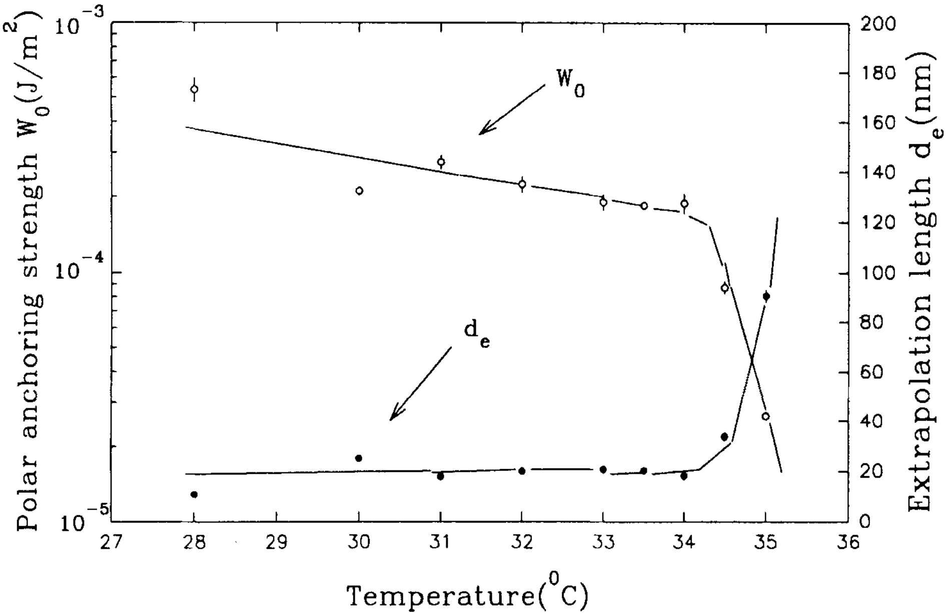

FIGURE 8 Polar anchoring strength and extrapolation length of rubbed PI-LB (5) versus temperature.

stress generated by the rubbing process apparently gives these backbones a small but finite angle with the surface plane and a finite pretilt angle is obtained.

This result is consistent with the pretilt generation model proposed by Geary *et al.*[19] However, it has to be mentioned here that it is hard to rub PI-LB films uniformly because of their ultrathin nature. Therefore, adjusting the rubbing strength is not a suitable way to control the pretilt angle for practical applications. We also find that hard rubbing can damage or even strip away PI-LB films. Controlling the pretilt angle of LC's aligned by PI-LB films is still a big challenge for the application of LB films in the LCD industry. Pretransitional birefringence has been used to probe the mechanism of an LC alignment for some years.[20–23] We report here the first surface induced pretransitional birefringence measurements of an LC cell with PI-LB films as alignment layers. Three samples were measured and results are presented in Figure 9. The alignment layers for these samples are a PI-LB (5) film, a rubbed PI-LB (5) film and a conventional spin-coated rubbed PI film. All three samples show qualitatively similar pretransitional behavior. The solid lines in Figure 9 are fitting curves using Eq. (3) derived from the Ginzburg-Landau theory of de Gennes.

Under the framework of de Gennes' theory,[24] the free energy density of a semi-infinite ($z \geq 0$) sample near the LC isotropic to nematic phase transition is

$$F = F_0 + \tfrac{1}{2}a(T-T^*)Q^2(z) - \tfrac{1}{3}bQ^3(z) + \tfrac{1}{4}cQ^4(z) + \tfrac{1}{2}L|\nabla Q(z)|^2 \tag{2}$$

Minimizing the total free energy with respect to the functional form of $Q(z)$ at fixed boundary condition $Q(0) = Q_s$, the analytic expression for total phase difference between

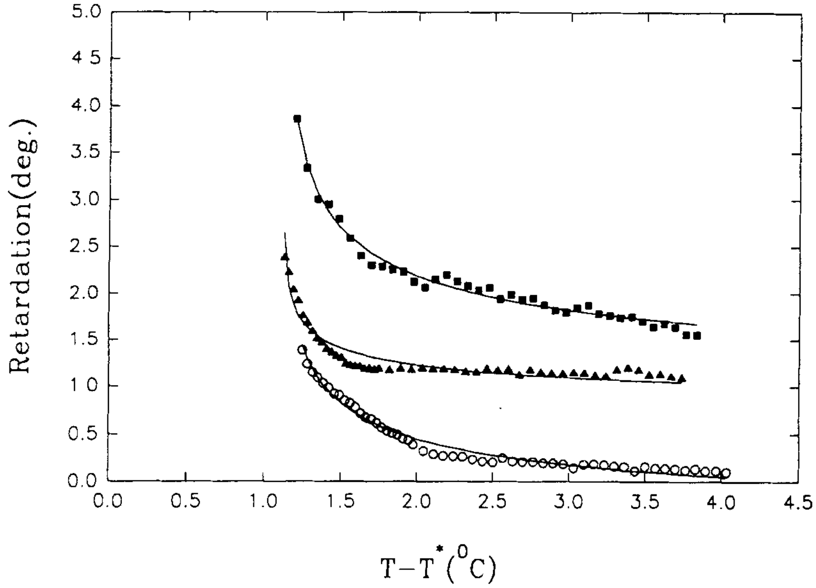

FIGURE 9 Pretransitional birefringence behavior of LC cells assembed with PI-LB (5) (○), rubbed PI-LB (5) (▲) and spin-coated rubbed PI (■) films. The solid curves are fitting curves using Equation (3). Both experimental and theory values of the middle curve were shifted upward one degree on purpose in order to avoid overlapping the bottom curve.

ordinary and extraordinary rays of light passing normally through the sample is[22]

$$\begin{aligned}\Delta\delta &= \frac{2\pi}{\lambda}(n_e - n_o)\int_0^\infty Q(z)dz \\ &= \frac{2\pi}{\lambda}(n_e - n_o)\left(\frac{2L}{c}\right)^{1/2}\ln P(T, Q_s) \\ &= \delta_0 \ln P(T, Q_s) \qquad (3)\end{aligned}$$

where

$$P(T, Q_s) = \frac{(F_1(Q_s)/Q_s^2)^{1/2} + (a(T - T^*)/2)^{1/2} + Q_s c^{1/2}/2}{(F_1(Q_s)/Q_s^2)^{1/2} + (a(T - T^*)/2)^{1/2} - Q_s c^{1/2}/2}$$

$$F_1(Q) = a(T - T^*)Q^2(z)/2 - bQ^3(z)/3 + cQ^4(z)/4$$

The parameters a, *b* and *c* for 5CB used for all fitting were $0.13 \times 10^6\,\mathrm{Jm^{-3}K^{-1}}$, $1.6 \times 10^6\,\mathrm{Jm^{-3}}$ and $3.9 \times 10^6\,\mathrm{Jm^{-3}}$ respectively and were obtained from the ref. 23. We selected Q_s, T^*, δ_0 and the background birefringence as adjustable fitting parameters to minimize the standard deviation.

Surprisingly, for all three cases, we found almost the same surface order parameters. The fitted values of Q_s were 0.38, 0.39 and 0.38 respectively for PI-LB (5), rubbed PI-LB (5) and spin-coated rubbed PI films. The fitting results for the PI-LB film and rubbed spin-coated PI film are fairly good. However, we notice that we can't fit both the high and low temperature regions for the rubbed PI-LB film. The magnitude δ_0 for the rubbed PI-LB film is 0.33, which is quite different from values 0.70 and 0.92 we got for PI-LB (5) and rubbed PI films respectively. They are supposed to be the same in the model because the same LC materials are used (see Eq. (3)). Note that the data for this film fit de Gennes' model rather poorly by comparison with the other two films. This may indicate that the surface order parameter of the rubbed PI-LB (5) film changes with temperature instead of being fixed as we have assumed or that the uniformity of the rubbed PI-LB films is poor. More detailed work on this is under way.

The nucleation of the nematic phase of homogeneous LC cells with PI-LB (5) as alignment layers was observed under polarized light microscopy as the cell cooled slowly. As described in ref. 19, "sheet nucleation" on the cell surface was observed as the nematic phase was entered. These uniformly-aligned nematic layers grew in thickness until they met in the center of the cell, producing a flaw-free aligned texture. This behavior is strongly distinguished from the evolution of the alignment texture of an LC cell associated with obliquely evaporated SiO_x alignment layers. In the latter situation, the nematic phase nucleates at numerous isolated sites in the bulk and at the surface. These spots grow and join together with a high density of director defects. Uniform alignment of the whole cell is reached only after the defects anneal out. The PI-LB alignment behavior belongs exactly to the first category in Table II ref. 19, i.e., sheet rather than point nucleation. On the other hand, obliquely deposited SiO_x alignment surfaces belong to the second category and are strong candidates for description by the Berreman model.[25] This is further substantiated by their failure to give pretransitional birefringence.[21]

Historically, the groove model was first proposed to elucidate the LC alignment mechanism for rubbed or polished solid surfaces[25]. Berreman, however, was well aware that surfaces having oriented long organic molecules may align by a different mechanism. Much evidence shows that the groove structures induced by the rubbing process play a minor role in LC alignment on rubbed PI or many other polymer surfaces.[19,27,28] An epitaxial growth model was subsequently proposed in which the interaction between oriented polymers and LC molecules is responsible for LC alignment.[19] The mechanism of alignment is analogous to epitaxial formation of conventional solid crystals except it is orientational rather than translational in nature. However, to some extent there is still confusion about these two models. All of the above experimental results taken collectively provide evidence to settle this issue in favor of epitaxial alignment. This conclusion is independent of any specific macroscopic model such as that of de Gennes.[24]

We studied the case of LB films with a varying number of layers deposited perpendicular to the direction of rubbing on a spin-coated rubbed PI film. We also found that three PI-LB layers ($\sim$ 13 Å) were required to switch the LC orientation from the rubbing direction to the PI-LB dipping direction.[7] Before deposition, well-defined grooves were detected by AFM. The depth of grooves was around 80 Å. As we mentioned above, 3 PI-LB layers were also required to achieve good LC alignment on ITO-coated glass. It seems that it also requires three PI-LB layers to screen the interaction between the LC and the polymer on spin-coated rubbed PI films. The groove structure was still observed by AFM. After 3 layer PI-LB film deposition this result shows, not surprisingly, that while the groove structure may aid alignment, it is not an intrinsic reason for LC alignment on a rubbed polymer surface. Note also that grooves on the rubbed polymer surface increases the interaction area between polymer and LC molecules in comparison with a PI-LB film with a flat surface. This could be the reason why the polar anchoring strength of a PI-LB film is lower than that of the rubbed PI film. Whatever the true effect of the grooves may be, when the interaction between oriented polymer and LC molecules is screened by three or more perpendicularly oriented PI-LB layers, the influence of groove structure is dramatically weakened.

4. CONCLUSIONS

Highly-ordered multilayer PI-LB films were successfully fabricated by the LB technique and showed a strong tendency to align liquid crystals. In order to achieve good alignment on ITO-coated glass, three or more PI-LB layers were necessary. It was also found that the anchoring direction of the LC was solely determind by the dipping direction of the uppermost PI-LB layer. This means that the alignment comes from short range molecular interactions. In our range of dipping speeds, the orientational order of our PI-LB films was almost constant. Unfortunately, they failed to align the LC with a pretilt angle. Rubbing was found to solve this problem in principle, but it was very hard to rub the whole region uniformly. The measured polar anchoring strength between the PI-LB film and 5CB is very strong ($\sim 3 \times 10^{-4}$ J/m^2 at 30°C). The alignment texture and pretransitional birefringence behavior of an LC cell with PI-LB alignment films clearly showed that the aligning LC layer grows epitaxially from the surface. In the case of PI-LB films deposited perpendicular to the rubbing direction of spin-coated rubbed PI films, results indicated that while the groove structure generated by the rubbing process was not the intrinsic reason for LC alignment on rubbed polymer surface, it may enhance the anchoring strength.

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