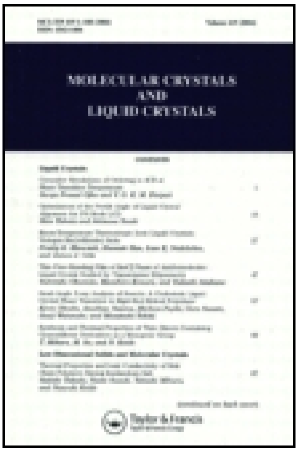

Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl19

# Topography of Thin Teflon Layers and Induced Orientation of Nematic Liquid Crystals

Marie-Pierre Cuminal [a] , Françoise Touhari [b] , Monique Brunet [a] & Gerard Cohen-Solal [b]

[a] Groupe de Dynamique des Phases Condensées- UMR 5581 Université Montpellier II, C.C.26 F-34 095, Montpellier Cédex, 05, France

[b] Laboratoire d´Analyse des Interfaces et de Nanophysique, UPRESA 5011 Université Montpellier II, C.C.82 F-34 095, Montpellier Cédex, 05, France

Version of record first published: 24 Sep 2006

To cite this article: Marie-Pierre Cuminal, Françoise Touhari, Monique Brunet & Gerard Cohen-Solal (1999): Topography of Thin Teflon Layers and Induced Orientation of Nematic Liquid Crystals, Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals, 333:1, 181-191

To link to this article: http://dx.doi.org/10.1080/10587259908026004

*Mol. Cryst. Liq. Cryst.*, Vol. 333, pp. 181-191
Reprints available directly from the publisher
Photocopying permitted by license only

Published by license under the
Gordon and Breach Science Publishers imprint.
Printed in Malaysia

# Topography of Thin Teflon Layers and Induced Orientation of Nematic Liquid Crystals

MARIE-PIERRE CUMINAL[a], FRANÇOISE TOUHARI[b], MONIQUE BRUNET[a]* and GÉRARD COHEN-SOLAL[b]

*[a]Groupe de Dynamique des Phases Condensées – UMR 5581 Université Montpellier II C.C.26 F-34 095 Montpellier Cédex 05 France and [b]Laboratoire d'Analyse des Interfaces et de Nanophysique – UPRESA 5011 Université Montpellier II C.C.82 F-34 095 Montpellier Cédex 05 France*

It has recently been shown that teflon (PTFE) films can be used as orientation layers to align liquid crystal molecules. In this paper we study the influence of the deposition parameters (pressure, speed and temperature) on the layer morphology given by both optical and atomic force microscopy. In addition, we have measured the surface pretilt angle induced on nematics by these films. It turns out that teflon gives a small pretilt, of the order of 0.3 degrees, at the surface. Although the topography of the deposited layer is seen to vary, the characteristic nematic textures do not vary with a variation of the deposition parameters.

Les couches minces en PTFE sont utilisées depuis peu comme couches d'orientation pour aligner les molécules de cristal liquide. Dans cette contribution, nous étudions d'une part l'influence des paramètres de dépôt (pression, vitesse et température) sur la morphologie des couches observées en utilisant les techniques de microscopie optique et de microscopie à force atomique. De plus, nous avons mesuré l'angle de pretilt induit sur les nématiques par ces films. Cet angle est faible, de l'ordre de 0.3°. Bien que la topographie des couches déposées varie lorsque les paramètres de dépôt changent, les textures du nématique ne changent pas.

*Keywords:* PACS. 68.55. -a – Thin film structure and morphology; PACS. 61.30 -v- Liquid crystals; PACS.42.79.Yd – Optical elements, devices, and systems

## 1 – INTRODUCTION

First described in 1990 by Wittmann and Smith [1], thin films of poly(tetrafluoroethylene) (PTFE), obtained by rubbing this polymer against a glass substrate at controlled temperature, pressure and speed, have since provoked considerable

---

* Corresponding author: Monique Brunet Fax: 04.67.14.46.37 E-mail: brunet @gdpc.univ-montp2.fr

interest because of their capacity to orient polymers and organic materials such as liquid crystals. This fluorinated polymer presents interesting characteristics such as high molecular weight, a rigid chain and a fusion domain above 300°C. Wittmann, Dietz and collaborators [1–4] have noticed the particular morphology of the deposited layers. They showed that the PTFE layer surface is not molecularly smooth and that the chains of the PTFE macromolecules are oriented parallel to the sliding direction and to the glass substrate. The use of these PTFE layers as alignment layers for liquid crystal cells is relatively recent and it is interesting to compare them with others layers commonly used such as evaporated SiO or rubbed polymers. The study of the topography of the surfaces is an important element for understanding anchoring and interfacial phenomena between the orientation layer and the liquid crystal molecules. To approach this problem we have made a comparative study of topography obtained with different deposition parameters. In the following we present observations obtained by optical microscopy and atomic force microscopy (AFM). We have also measured the pretilt angle of the director, as induced by these PTFE layers, using an optical setup [5] based on the crystal rotation method. Our aim was to correlate the surface morphology of deposited teflon with the induced oriention of the nematic director.

## 2 – SURFACE MORPHOLOGY

### 2.1 – Deposition technique

The deposition of the thin PTFE layer is simple and entirely mechanical. The principle is the same as the one presented by Wittmann and al. [1]. A cylindrical PTFE bar is placed on a plane substrate at controlled and constant temperature and pressure, and rubbed only once against the substrate at controlled and constant speed. Thus a thin layer of PTFE is directly transfered onto the support. This method has been established in our laboratory by S. Quentel [6]. The substrate used is a glass plate (1.1mm thick and 2 × 3 $cm^2$ surface) from Balzers coated with a thin ITO (Indium Tin Oxide) layer, carefully cleaned and dried, like the ones used to make liquid crystal displays. In order to compare the influence of preparation parameters on the layer morphology the deposition temperature can be chosen between 150°C to 300°C, the pressure applied on the PTFE bar can vary from $10^6$ Pa to $5.10^6$ Pa and the deposition speed can be changed from 0.1 $mm.s^{-1}$ to 1.5 $mm.s^{-1}$. The laboratory device is schematically represented in figure 1.

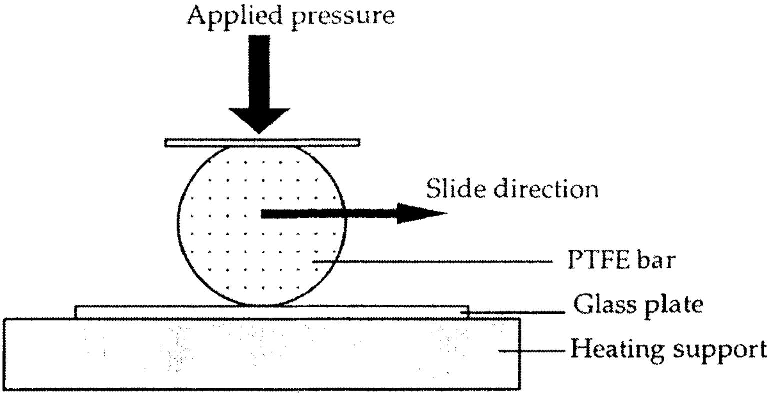

FIGURE 1 Schematical presentation of the PTFE deposition device

## 2.2 - Optical microscopy

To observe the morphology of our deposited PTFE layers we have used optical microscopy in phase contrast. Using a Heine condensor we can obtain different sorts of lighting and so can pass continously from phase contrast to dark field. Dark field observations allow a surface morphology evaluation and a comparative study of different layers. We have investigated PTFE films grown under different deposition conditions. We notice that the beginning and the end of the PTFE coatings are quite disturbed zones, probably due to transition from static to sliding friction condition and associated to pressure and temperature variations on the PTFE bar. However, the PTFE layers appear relatively uniform everywhere else, showing stripes of various width and spacing, parallel to each other and to the sliding direction.

These optical observations show, as already emphasized by Dietz et al. [3], that the most influential deposition parameter is the temperature, cf. figures 2 and 3 presenting two optical microphotographs of PTFE layers obtained with two different deposition temperatures. In fact, when the deposition temperature increases, from 150°C to 300°C, the PTFE stripes appear more regular and less spaced from each other. Moreover, the fraction of covered surface increases with higher temperature, when the stripes become broader. Increasing the applied pressure on the PTFE bar from $2.5\ .10^6$ Pa to $5\ .10^6$ Pa also increases the layer continuity as observed with atomic force microscopy (photographs of figures 4 and 5). On the other hand, no variation was observed on the layer morphology when increasing the speed from $0.1\ \mathrm{mm.s^{-1}}$ to $1.5\ \mathrm{mm.s^{-1}}$.

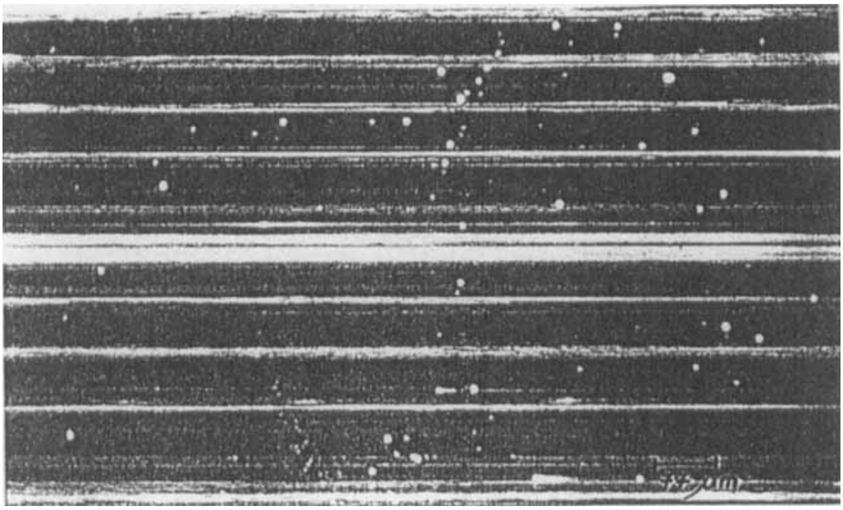

FIGURE 2 Dark field optical microscopy of a PTFE layer after deposition at temperature 230°C, speed 0.6 mm.s$^{-1}$ and pressure $P_2 = 5.10^6$ Pa

## 2.3 – Atomic force microscopy

The topographical analysis of these PTFE layers has been made by atomic force microscopy (A.F.M.). The advantage of the contact mode used is to allow a nanometric resolution which is necessary to characterize PTFE films. However, this implies that important forces should be applied to the cantilever to maintain it in contact with the surface in spite of the strong repulsive forces occurring between the tip and the sample. As this could lead to a deformation of the sample surface, low forces have been used.

Besides, if the PTFE stripes are not connected to the ITO surface the tip can dissect and sweep them which gives fibers presenting a zigzag structure such as observed by Dietz and al. [3].

On each sample several images were made at large scale (30 μm) on different places in order to choose one image representative of all the surface. Images presented here are 10 μm × 10 μm large and were all acquired using the same tip. Acquisitions were made in air using microfabricated cantilevers with a small spring constant (0.16 N.m$^{-1}$) supporting a silicon conical tip with curvature radius less than 10 nm manufactured by Park Scientific Instruments. Since the film never covers the substrate completely, the PTFE thickness can be determined on height profiles relative to the substrate.

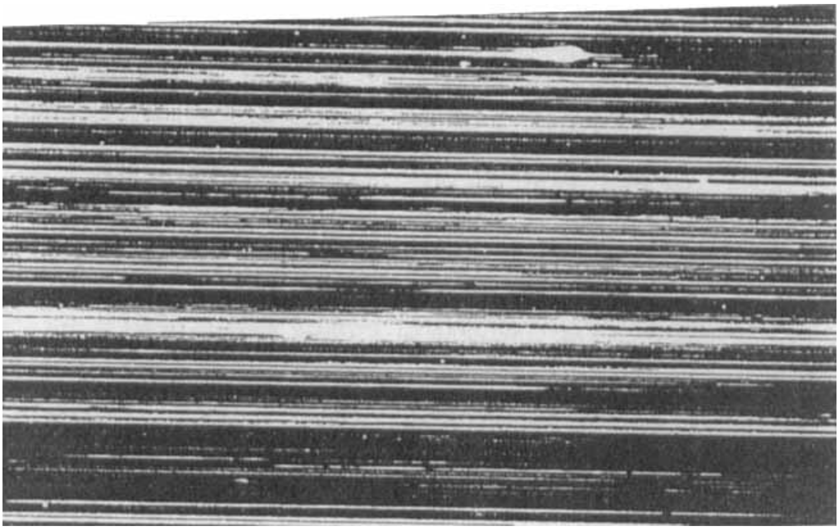

FIGURE 3 Dark field optical microscopy of a PTFE layer after deposition at temperature 300°C, speed 0.6 $mm.s^{-1}$ and pressure $P_2 = 5.10^6$ Pa

The observation of a coating strongly depends on the substrate roughness. Indeed, in the case of ITO substrate the oxide particles are approximately 10 nm large and, as a consequence, are visible on the AFM image under the PTFE stripes. This of course limits the resolution which could be obtained if the substrate were smooth. As the PTFE coating is not uniform the substrate is visible between the stripes. Thus if the PTFE fibers are thin (a few angstroms) the presence of the oxide particules hides them. However, the presence of PTFE fibers can be guessed from the AFM images because of the significant stripes but they are so thin that if one makes a height profile by using the software the fibers cannot be distinguished from the oxide particules.

The influence of the pressure applied on the PTFE bar on the coating is clearly shown on figure 4 corresponding to $P_1 = 2.5\ 10^6$Pa and figure 5 for which $P_2 = 5.10^6$ Pa. For each of these figures we report the width of the cross section perpendicular to the orientation of the film which shows that the height of the film is not homogeneous. For the pressure $P_1$ the ITO surface is visible on a large part of the image due to the fact that the PTFE stripes are quite thin and spaced. When increasing the pressure from 2.5 $10^6$ Pa to $5.10^6$ Pa the coverage is more important. The spacing between the stripes varies from 0.2 to 0.8 μm for 2.5 $10^6$ Pa, the fiber width goes from 0.2 μm to 0.09 μm but the presence of smaller fibers between larger ones can be guessed from the AFM image. The average

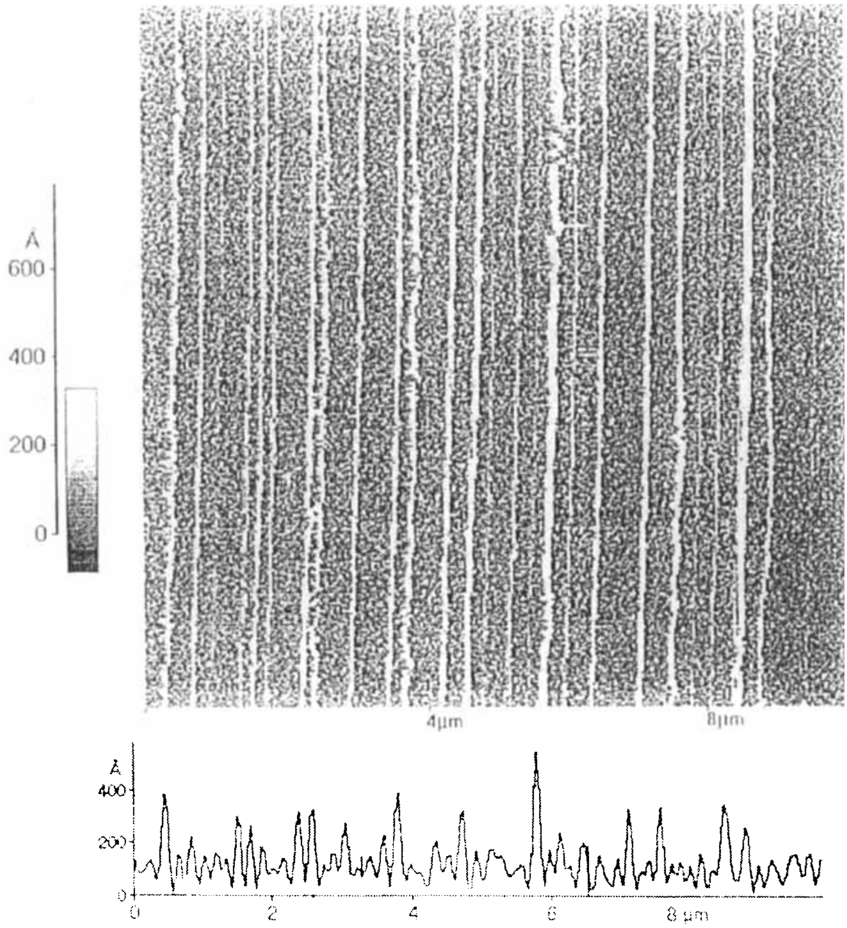

FIGURE 4 Atomic force microscopy image and corresponding height profile of a cross section of a PTFE layer. Same temperature and speed. The pressure is $P_1 = 2.5.10^6$Pa

height measured on these scans is 12 nm for $P_1$ and 65 nm for $P_2$. Moreover, for this latter pressure one observes large stripes (from 0.5 to 0.9 µm) which are sometimes linked between them by small fibers. In figure 6 we represent the height histogram for these samples. It shows that in the case of pressure $P_1$ one has a relatively narrow distribution with only one characteristic height around 7 nm whereas for pressure $P_2$ the height has increased to 40 nm with a smaller second peak at about 75 nm. This demonstrates the influence of the pressure on the fiber thickness: when increasing the pressure larger stripes are deposited on the substrate.

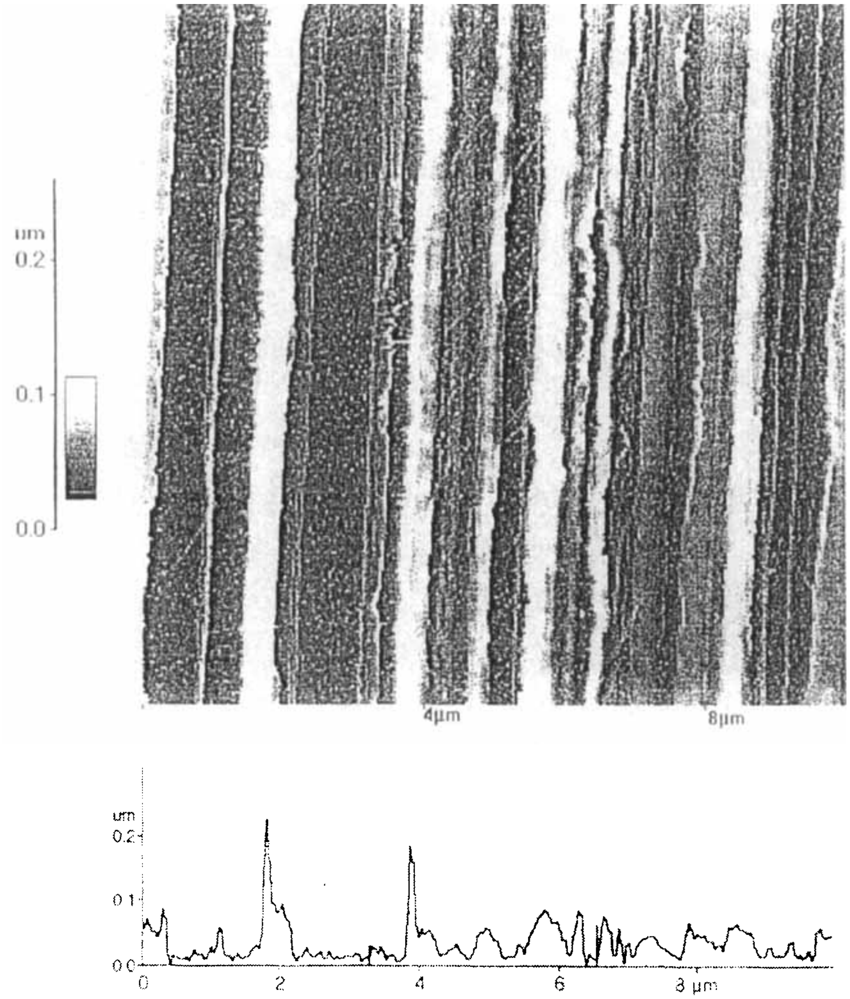

FIGURE 5 Atomic force microscopy image and corresponding height profile of a cross section of a PTFE layer. Same conditions, excep that the pressure has been increased to $P_2 = 5.10^6$ Pa

The effects of temperature and pression are correlated. However, AFM observations on two PTFE layers obtained with different deposited parameters: 2.5 $10^6$ Pa and 300°C for the first one and 5 $10^6$ Pa and 230°C for the second one, show that even if the pressure is greater, the fibers are thinner (12 nm for the first sample and 8 nm for the second one) because of the lower temperature.

Increasing the deposition temperature or pressure increases the layer continuity in form of the number and the width of PTFE stripes. However, as was already pointed out by Dietz et al.[3], the temperature is the most important deposition parameter. In the case of layers deposited at 230 °C the surface is less covered and presents very thin fibers with an average height of 7 nm whereas for 300 °C the average is 55 nm.

Our observations show a characteristic surface topography very different compared to the one after obliquely evaporated silicon oxide (SiO) [7–9]. PTFE layers are formed with stripes parallel to each other and to the sliding direction whereas evaporated SiO layers are more uniformly rough. Nevertheless these PTFE layers can be used as alignment layers to orient liquid crystals as well as obliquely evaporated SiO or rubbed polymers, showing typical liquid crystal textures with a fibrous aspect. Although the layer topography varies with deposition parameters, we did not observe differences in the nematic textures obtained.

## 3 – STUDY OF THE NEMATIC LIQUID CRYSTAL ORIENTATION

### 3.1 – Optical observations of cells

To study the quality of alignment of liquid crystals on the teflon coating, we have prepared cells made with two substrates coated with PTFE, created by sliding directions antiparallel, spaced with four mylar spacers 12 µm thick and glued with Norland UV91. The cell gap was measured with a spectrometer and the cells filled with a liquid crystal in the isotropic phase. We have used liquid crystal materials from E. Merck such as ZLI-3219 and ZLI-4389 which are nematic at room temperature.

When observed with polarising microscope, the cells appear relatively well oriented with extinction when the sliding direction is parallel to one of the crossed polarizers. Using a quarter wave plate we can determine the director orientation of the liquid crystal, which is found to be parallel to the sliding direction. The cells are characterized by a fibrous texture (corresponding to the surface topography) different from the one observed with evaporated SiO or rubbed polymers. We have noticed that the molecular orientation has better homogeneity when the deposition temperature lies over 250°C. We observe a preferential orientation of the liquid crystal director in spite of the dimensions of the surface irregularities (i.e. the PTFE stripes and their distribution) over a much larger scale than molecular dimensions. Nevertheless, this effect is in agreement with the theory of de Gennes and Berreman [10–12] who associate the anchoring energy with the surface morphology.

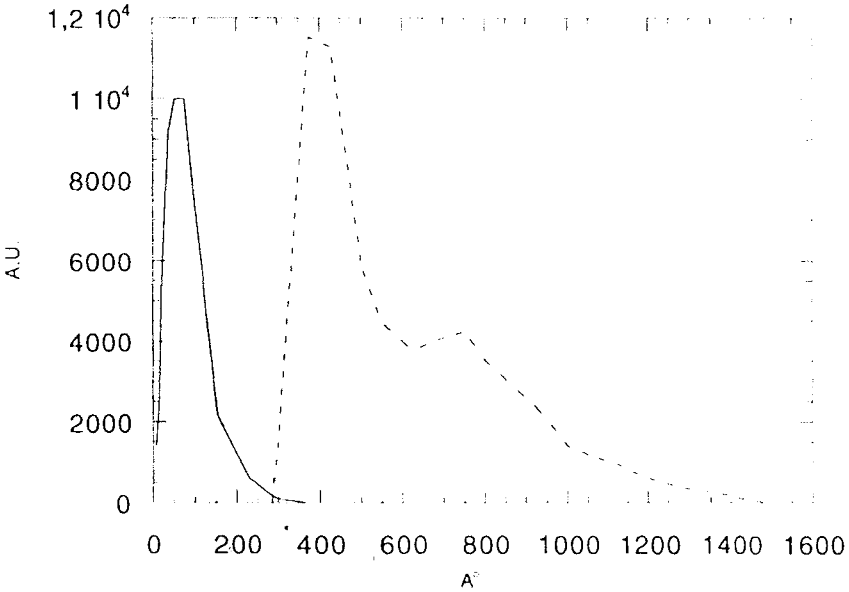

FIGURE 6 Height histograms for the samples in figures 4 and 5. Full line corresponds to $P_1 = 2.5.10^6$ Pa, dashed line to $P = 5.10^6$ Pa

### 3.2 – Pretilt angle measurements

The pretilt angle is the angle between the liquid crystal director and the cell surfaces. This angle is induced by the alignment layer which causes a preferential orientation on the cell surfaces and thereby in all of the sample if this is not too thick. The pretilt angle strongly influences the characteristics of liquid crystal devices and has to be measured with good reproducibility in order to understand anchoring effects. In a previous publication [5], we have shown that the pretilt value strongly depends on the chosen couple liquid crystal/orientation layer. In fact, we obtained very different pretilt values for one compound on different layers and also for different compounds on the same layer.

To measure the pretilt we have used our laboratory optical setup already described [5] and schematically represented in figure 7. Based on the crystal rotation method, the originality of this setup is to allow measurements with the same precision of ±0.3° for any value of the cell thickness. The liquid crystal cells are prepared as described before. The director is supposed to have a homogeneous distribution through the cell, forming an uniaxial single crystal, the optic axis of which makes a pretilt angle $\alpha$ with the plane of the substrates. The crystal rotation method is based on the measurement of the cell transmission between two crossed polarizers, oriented at 45° from the incident plane, as a function of the incident angle on the cell, wich is rotated as shown in the figure. Using the transmission curve we can calculate the pretilt from the known values of the ordinary and the extraordinary refractive indices of the liquid crystal.

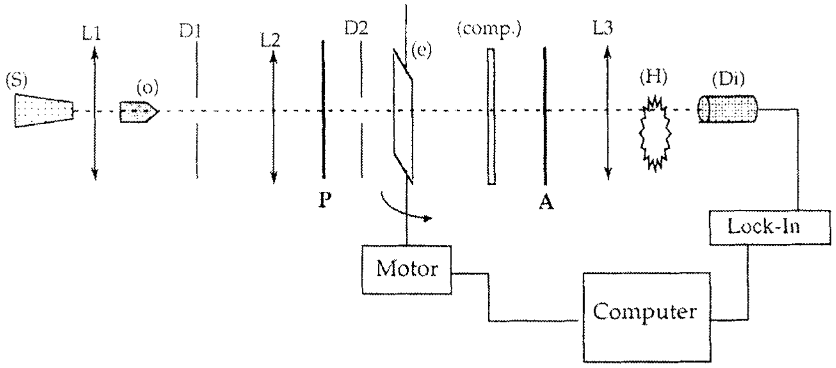

FIGURE 7 Scheme of the experimental optical set-up: (S) is a white light source, (e) the LC sample, (o) microscope objective, (H) chopper, (Di) detection diode, (comp) compensator, L1, L2, L3 lenses, D1 and D2 diaphragms, P represents the polarizer and A the analyzer

The pretilt angles were measured for different deposition conditions. In table I, the deposition temperature was allowed to vary whereas the pressure applied on the PTFE bar is $5.10^6$ Pa and the deposition speed is 1.5 $mm.s^{-1}$. Two nematic compounds at room temperature have been compared. In tables II and III, the deposition temperature is 300°C at variable pressure and speed.

TABLE I Pretilt angles induced by PTFE layers deposited at various temperatures for two nematic liquid crystal compounds

| *Deposition temperature* | *ZLI-3219* | *ZLI-4389* |
|---|---|---|
| 190°C | 0.3° | - |
| 260°C | 0.2° | 0.3° |
| 300°C | 0.4° | 0.5° |

TABLE II Pretilt angles induced by PTFE layers deposited at two different speeds, at the same temperature, 270°C

| *Deposition speed* | *ZLI-3219* |
|---|---|
| 0.6 $mm.s^{-1}$ | 0.4° |
| 1.5 $mm.s^{-1}$ | 0.4° |

TABLE III Pretilt angles induced by PTFE layers deposited with two different pressures, at constant speed (0.6 $mm.s^{-1}$) and temperature (270°C)

| *Deposition pressure* | *ZLI-3219* |
|---|---|
| $2.5.10^6$ Pa | 0.2° |
| $5.10^6$ Pa | 0.4° |

The results show that, in spite of different surface topography and alignment quality, teflon induces a pretilt which is quite small, of the order of 0.3°, independent of deposition parameter variations.

## 4 - CONCLUSIONS

Despite their particular surface morphology, PTFE layers can align liquid crystals equally well as can SiO or rubbed polymers. Topographical observations in optical and atomic force microscopy show a certain influence of the deposition parameters on the PTFE surface topography and, in particular, the importance of the temperature on the deposited layer. Pretilt angles measured are systematically small, around 0.3°, independent of the deposition parameters and the liquid crystal material.

### *References*

[1] J.C. Wittmann, P. Smith, *Nature* **352**, 414 (1991).

[2] F. Motamedi, K.J. Ihn, D. Fenwick, J.C. Wittmann, P. Smith, *SPIE* **1665**, 194 (1992).

[3] Dietz P., Hansma P.K., Ihn K.J., Motamedi F., Smith P., *J. Mat. Sci.* **28**, 1372 (1993).

[4] S. Meyer, Thesis, University Strasbourg (1995).

[5] M.P. Cuminal, M. Brunet, *Liq. Cryst.* **22(2)**, 185 (1997).

[6] S. Quentel, Thesis, University Montpellier (1993).

[7] J.L. Janning, *Appl. Phys. Lett.* **4**, 173 (1972).

[8] W. Urbach, M. Boix and E. Guyon, *Appl. Phys. Lett.* **25**, 479 (1974).

[9] M. Monkade, M. Boix and G. Durand, *Europhys. Lett.* **5(8)**, 697 (1988).

[10] P.G. de Gennes and J. Prost, *"The Physics of Liquid Crystals"*,edited by Oxford Science Publications, (Oxford 1993).

[11] D.W. Berreman, *Phys. Rev. Lett.*, **28**, 1683 (1972).

[12] D.W. Berreman, *Mol. Cryst. Liq. Cryst.*, **23**, 215 (1973).