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Rubbing-induced surface textures in nematic MBBA layers and their behaviour under applied d.c. or a.c. voltages

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Surface irregularities (for example, rubbing-induced domains, and bumps) have been observed in MBBA nematic cells after special treatment of the electrodes using a conventional soap. Various techniques such as polarizing microscopy, the shadowgraph technique and computer processing of the images have been used. The behaviour of the rubbing-induced domains under a.c. or d.c. electric fields has been studied. A new flexoelectric effect has been observed leading to the division of the rubbing-induced domains. A simple model has been proposed (a modification of the Saupe model), which explains the structure and division of the rubbing-induced domains.

1. Introduction

The surface orientation, anchoring and homogeneity of liquid crystals, and in particular of nematic liquid crystals, is of crucial importance for the successful operation of liquid crystal displays. The problem of liquid crystal orientation has been recognized to be of utmost importance ever since the discovery of liquid crystals in 1888. Amongst the early research the work of Zocher and Coper should be mentioned [1, 2], in which the orientation of a liquid crystal was realized by rubbing the glass plates with diamond powder or cerium oxide in one direction, then cleaning and baking them to remove the organic materials. Many researchers [3–9] used this procedure for the treatment of glass plates. The second important way to orient liquid crystals by long-range elastic forces is the evaporation method, first proposed by Janning [10]. This consists of the oblique evaporation of silicon or other oxide (or a metal) on the glass slides confining the liquid crystal. Depending on the angle of evaporation, one can achieve planar or tilted orientation of the liquid crystal [11, 12]. Finally, nematics can be oriented on surfaces previously covered with various submicrometer or micrometer periodic orienting gratings, with various periodicity and shapes such as rectangular, trapezoidal, sawtooth-like, or semi-cylindrical [13–18].

The surface-generated orienting forces acting on the liquid crystals result not only from the elastic interactions arising from the anisotropic elasticity of the liquid crystal, but also from physical-chemical causes such as

hydrogen bonding, van der Waals interactions, and dipolar interactions. It has long been well known that these forces can be crucial for the orientation of the liquid crystal, not only for non-treated or semi-free nematics, but also for surfactant-treated surfaces [19–25]. The surface behaviour of liquid crystals, and in particular of nematic liquid crystals, can be explained by such important phenomena as wetting, partial wetting and drying, and including the well known Young's law and its modifications [26, 27]. The first step in this direction was made by Creagh and Kmetz in 1973 [6] who proposed a simple rule for the orientation of a liquid crystal covering a glass plate, involving the surface tension of the liquid crystal γ_L and the critical surface tension of the solid surface γ_c [28]. It is usually accepted that when the liquid crystal wets the solid, the orientation is planar and when the wetting is partial, the orientation of the liquid crystal on the supporting surface is tilted or homeotropic [29]. However, there are examples when the rule of Creagh and Kmetz does not hold [23, 24, 30].

The three main treatments of the glass plates supporting the liquid crystal described already—rubbing, oblique evaporation of an oxide or metals and the various surfactant covers—have been used in some cases simultaneously in order to achieve a desired orientation of the liquid crystal layers, displaying new electro-optical effects. For instance, rubbing of the glass plates followed by a surfactant deposition has been performed by Ogawa *et al.* [31] and by Bryan-Brown *et al.* [32] in order to obtain high-tilted nematic layers.

Many years ago, one of the authors (H.P.H.)

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discovered that a nematic liquid crystal confined between two glass plates treated using a conventional soap exhibits weak polar and azimuthal surface anchoring, and under the application of a d.c. voltage displays new surface-induced flexoelectric domains [33]. In the early work the glass plates were washed in a bath of water and soap. Later we utilized a simpler and reproducible technique consisting of an initial rubbing of the glass plates with a diamond paste spread on a cloth, cleaning by acetone or another organic liquid, a second rubbing with a dry conventional soap in the same direction followed by cleaning with a cloth, with rubbing in the same direction. In this way weakly anchored nematic layers were prepared and used either for the further study of the surface-induced flexoelectric domains [33–37] or for the study of various electro-optic effects in weakly anchored nematics or large pitch cholesterics [38–42]. We have observed recently in such nematic cells rubbing-induced surface domains with a period typically in the range 5–10 μm , irrespective of the thickness of the liquid crystal layer under study. Such domains can be observed when the focusing of the polarizing microscope is on the glass plate and the light transmitted through the domains is polarized in the direction perpendicular to the domains.

The rubbing-induced domains, and more precisely the rubbing-induced surface textures, contain many defects such as domain walls, disclinations and bumps which can be effected easily under the application of d.c. or a.c. voltages. The behaviour of the nematics was very variable, depending on the frequency of the applied electric field. For instance, the application of a high frequency electric field with a frequency above the cut-off for charge relaxation (in our experiment between 5 and 200 kHz) orients the MBBA nematic layer under study and leads to a clear separation of the θ -polar or zenithal deformations and ϑ -azimuthal deformations. Under the application of a d.c. voltage, however, we have observed a doubling of the domain period. Sometimes, depending on the initial conditions, we have observed the formation of a complex honeycomb texture, which at a low voltage (below 4 V) was static, while at a higher voltage became electrohydrodynamic. Here it should be noted that various rubbing-induced surface textures have been obtained and studied over the last thirty years as part of the intensive investigation of liquid crystals either for fundamental research or for practical applications [3, 7, 19, 43–49]. Since these domains usually disturb the initial orientation of the liquid crystal and reduce the homogeneity of liquid crystal displays, it is very important to understand the causes for their appearance and their behaviour under the application of d.c. or a.c. voltages. On the other hand, the rubbing-induced domains allow us to obtain

and study a new manifestation of the well known flexoelectric phenomenon in nematics.

The study of surface-induced textures was performed using a polarizing microscope equipped with a charge-coupled device (CCD) camera and a microcomputer. The most important images were analysed by the optical Fourier method. This method allows a deeper understanding of the optical or electro-optical changes in the liquid crystal orientation.

This paper is organized as follows. First, we describe experimental observations for the liquid crystal MBBA accompanied by photomicrographs taken for several liquid crystal cells with various orientations and anchoring. The study was chiefly performed under the application of a d.c. voltage. Additionally, for clarification of the liquid crystal orientation, we used a high frequency electric field. Further, dividing the most important images into 512×512 pixels, we studied them by optical Fourier analysis using a computer. Finally, we explain qualitatively our experimental results, including the Fourier analysis data, giving a simple model, that clarifies the doubling of the periodicity of the observed surface-induced domains, regardless of the value of the initial period.

2. Materials, sample preparation and methods of study

The experimental results described in this paper have been obtained using conventional sandwich cells filled with the liquid crystal MBBA (*p*-methoxybenzylidene-*p*-*n*-butylaniline) which has a clearing temperature between 42 and 45°C. The glass plates confining the liquid crystal were semitransparent and conductive and separated by two isolating sheets of Teflon or another polymer with a thickness between 10 and 50 μm . The orientation of the liquid crystal was achieved by the conventional rubbing technique. The glass plates were initially rubbed manually in one direction with a diamond paste spread on a cloth (grain size around 1 μm), then cleaned in the standard way, followed by a second rubbing using the conventional soap in the same direction and then cleaned again by rubbing with a cloth in the same direction. This simple procedure is shown schematically in figure 1. We note that after these treatments the thickness of the soap layer is very small, probably below 0.1 μm , and is not visible either by the naked eye, or under the microscope. In effect, the thickness of the soap layer cannot be greater than the depth of the deepest grooves which is in the range 0.2–0.5 μm .

The soap-treated glass plates, as noted previously, considerably influence the anchoring and the orientation of the MBBA nematic layers under study [33–42]. Such glass plates usually afford high-tilted MBBA

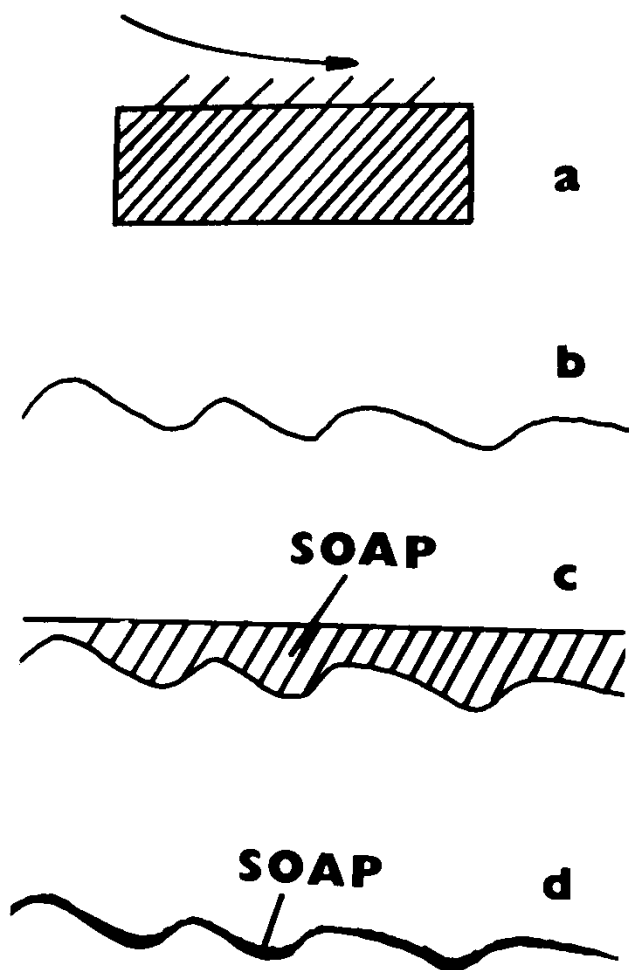


Figure 1. Rubbing technique with soap: (a) the direction of rubbing determines the sign of the tilt of the director at the boundary for the case of a hard material; (b) asymmetrical grooves after rubbing with a diamond paste; (c) a thick deposition of the soap after rubbing in the same direction; (d) a thin deposition of the soap after rubbing with a cloth in the same direction.

layers when the rubbing of the two glass plates is in two opposing directions, or nearly planar layers when the rubbing is in just one direction. The slight tilt of the nematic layer, relative to the normal to the glass plate, is due to asymmetrical grooves produced along the rubbing direction (figure 1). This suggestion is in agreement with the proposed model for obtaining a tilted orientation of the liquid crystal according to Nakamura [9], and confirmed by others [18, 50, 51]. Another, although less probable, mechanism could be the initial slight inclination of the aliphatic chains of the soap molecules and the existence of nanometer holes in the soap layer [52, 53].

Strong planar anchoring of the MBBA cells was achieved by rubbing a preliminarily spread thick

polyvinyl alcohol (PVA) layer using the procedure, described elsewhere (see, for example) [54]. The sandwich liquid crystal cells prepared in this way were hermetically sealed, to avoid impurities entering from outside [55, 56]. The soap deposition did not change the electrical properties of the conductive glass plates. The resistance of the conductive layer covering the glass plates was in the range 20–60 ohms and did not change after the soap deposition. Part of the liquid crystal cells consisted of only one glass plate treated by the rubbing technique described and covered with a thin layer of liquid crystal, several microns thick. The surface textures were observed under a polarizing microscope with a magnification between 6.3×12 and 20×12 . The nematic MBBA layers were excited with a d.c. voltage between 1 and 10 V. Some of the MBBA cells were further investigated under a high frequency electric field of frequency 200 kHz and amplitude between 1 and $15 V_{\text{rms}}$.

Most of the surface textures were studied as follows. Each of the selected photomicrographs was divided into 512×512 pixels and analysed by a personal computer (Pentium 133 MHz/32 microB RAM). The area of each pixel depends on the magnification of the polarizing microscope and the attached additional optical technique. For example, the dimensions of the pixel varied between 1×1 and $0.5 \times 0.5 \mu\text{m}^2$. This accuracy was relatively good since it corresponds to the optical resolution of the polarizing microscopes used in our experiment.

A significant part of the experimental results was obtained using the shadowgraph method [57] (in some cases this is called the method of optical contrast).[†] In this method, the analyser was not used. Another part of the experimental results was obtained using the conventional birefringent optical method utilizing polarizer and analyser. During the optical observations, a collimated beam of light, polarized perpendicularly to the initial alignment of the director, traverses the cell along the direction of the electric field. The resulting image is acquired with a charge-coupled device (CCD) camera attached to a microscope and digitized by a frame grabber with a spatial resolution of 512×512 pixels, 256 grey levels, which corresponds to 8 bits. The digitized images are stored and processed on a computer [56].

3. Experimental results

3.1. Polarizing microscopy

In order to clarify the nature of the surface textures, we prepared a number of semi-free MBBA samples. They consisted of one glass plate, prepared using the

[†]Part of the experiment was performed at the Bayreuth University, Germany, the other at the Institute of Solid State Physica, Bulgarian Academy of Sciences, Bulgaria.

rubbing technique described, over which a thin layer of the liquid crystal was spread. One example is shown in figure 2. This is a photomicrograph of a rubbed glass plate, part of which was not covered by the liquid crystal (the lower part of the photomicrograph). The other part of the glass plate was covered with a thin (a few microns) layer of the nematic MBBA (the upper part of the photomicrograph). Sometimes, the thickness of the liquid crystal was larger, in the range 5–10 μm . One can see on the photomicrograph regular scratches produced by the rubbing process and which determine the formation of the initial liquid crystal texture. The periodicity of the scratches and of the liquid crystal domains was between 15 and 20 μm .

Two more glass plates, treated using soap and covered with a thin MBBA nematic layer are shown in figures 3(a) and 3(b). It is evident that the liquid crystal textures are very similar, with a periodicity between 8 and 10 μm . On the other hand, it is clear that the soap treatment of the glass plates affords the

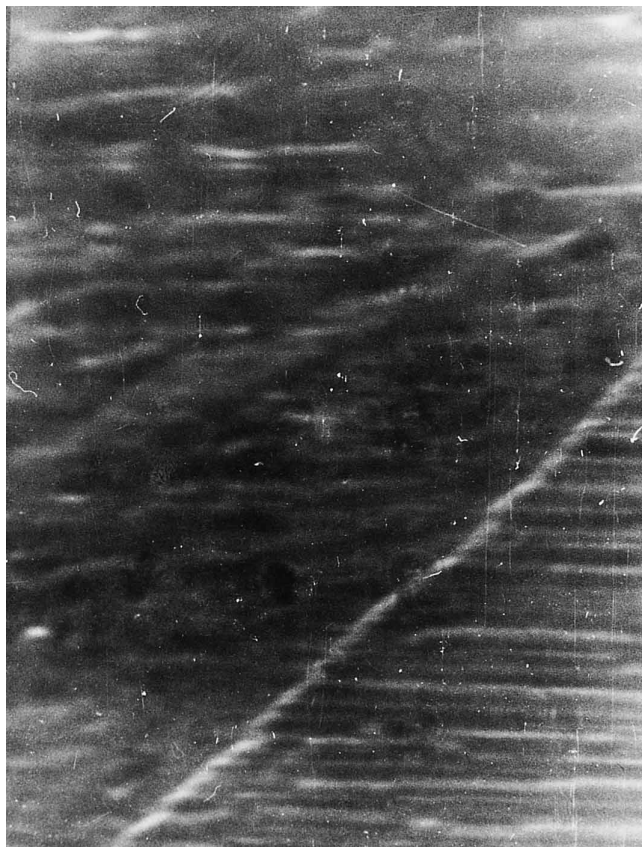
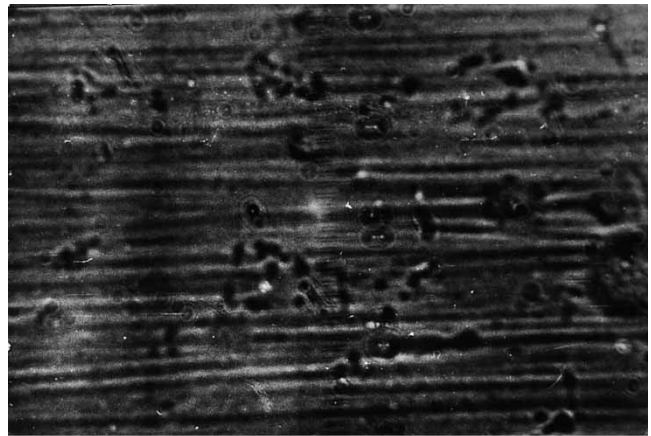


Figure 2. Photomicrograph of a glass plate prepared using the rubbing technique without soap. The upper part of the glass plate is covered with a thin layer of the nematic MBBA at room temperature. The polarizer is perpendicular to the grooves, and there is no analyzer. The short side of the photomicrograph corresponds to 328 μm .



(a)



(b)

Figure 3. Photomicrographs of two glass plates completely covered with a thin layer of the nematic MBBA at room temperature. The glass plates (a) and (b) were treated with soap according to the procedure described. The polarizer is perpendicular to the grooves, and the analyser has been removed. The short side of the photomicrograph corresponds to 328 μm .

development of rubbing-induced domains with a smaller period (compare 8–10 μm versus 15–20 μm). This experimental fact is also confirmed by the Fourier analysis (FA) results described later. The FA results reveal the range of wave numbers of the periodicities produced by the rubbing process, and clearly visualized by the soap deposition. Further, we have prepared a nematic MBBA cell of thickness 50 μm and having asymmetrical strong-weak anchoring. The strong planar anchoring was achieved after rubbing the thick PVA layer covering

one of the two glass plates confining the liquid crystal. The weak anchoring of the nematic was achieved using the soap treatment of the other glass plate, according to the procedure described previously. The nematic MBBA cell was subjected to a rectangular pulse voltage with a duration of 30 s and an amplitude of approximately 0.14 V, varying step by step from 1.18 to 6.83 V. Some selected surface textures are shown in figures 4(a) to 4(d) caption. These experimental results show, firstly, that a volume flexoelectric instability appears which has a fixed period and a threshold voltage (more precisely, optical threshold voltage) of 2.45 V; secondly, that the period of the rubbing-induced

domains decreases by about half. This is also evident from the FA results.

The surface-induced deformations, and in particular the type of the rubbing-induced domains, strongly vary from place to place in the cells. This can be seen from the photomicrographs shown. It is clear that the surface-induced texture is connected with the nature of the surface topography and in particular with the existence of scratches, typical of the rubbing process. The flexoelectric changes of these textures under the application of a d.c. voltage are connected not only with bulk interactions, but also with surface interactions. The latter depend crucially on the anchoring of the liquid

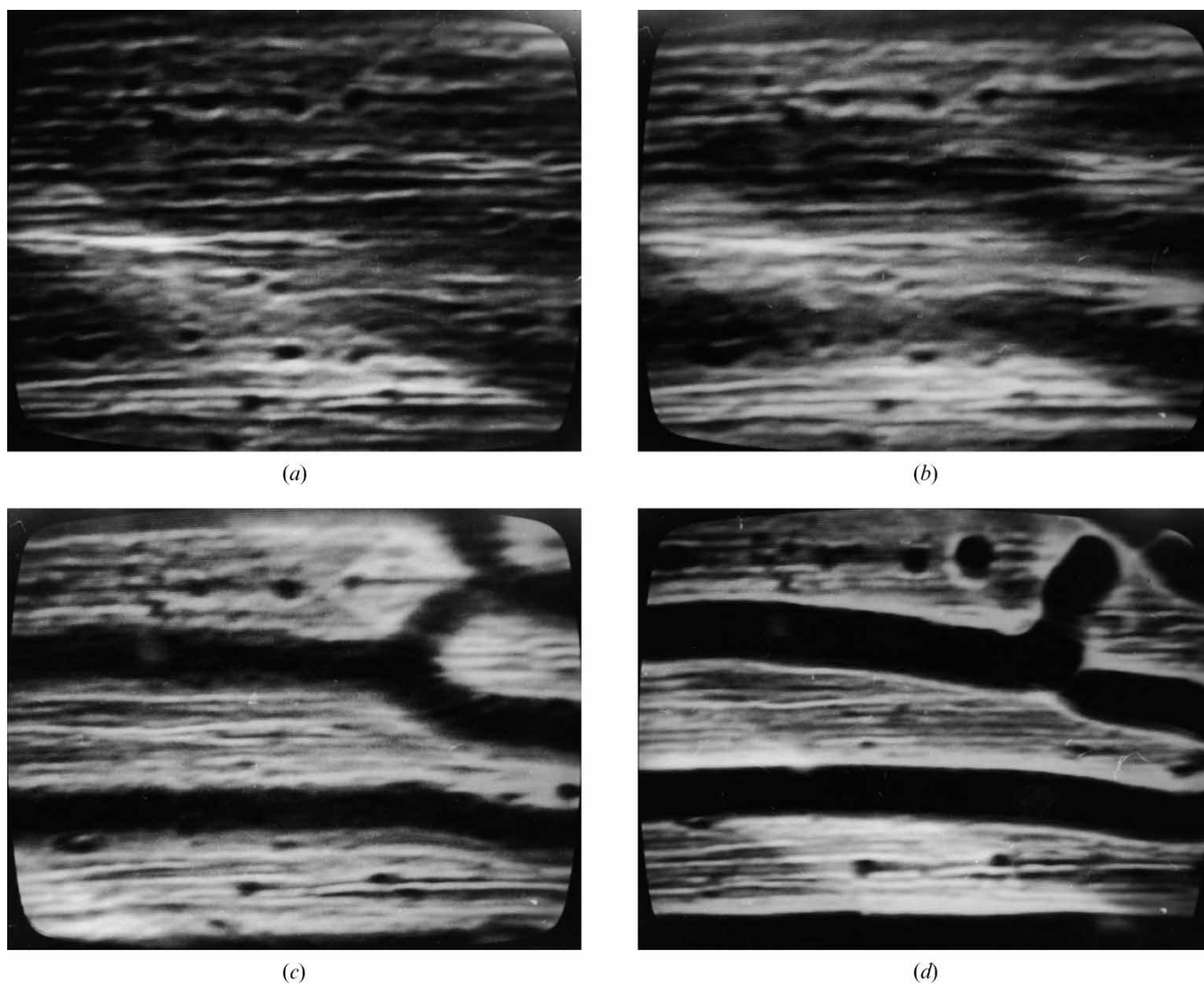


Figure 4. Photographs taken of a monitor showing longitudinal surface-induced flexoelectric domains and rubbing-induced domains in an asymmetrical strong-weak anchored MBBA nematic layer with a thickness of $50\ \mu\text{m}$ at room temperature under an applied d.c. voltage. The polarizer is perpendicular to the domains, and the analyser has been removed. The short side of the photograph corresponds to $256\ \mu\text{m}$. The applied d.c. voltage was (a) 1.18 V, (b) 2.71 V, (c) 4.36 V, (d) 5.46 V.

crystal varying from one place to another in the liquid crystal layer.

It should be mentioned that it is very difficult to observe the changes of the surface textures within time-intervals of seconds or even minutes. There are several ways to observe these changes. First, they can be seen if one studies the changes in the surface structures frame by frame; this is a very long and difficult process. The second way is to compare the images after a long period of time, e.g. compare the surface textures shown in figures 4(a) to 4(d). This shows only the final differences. One cannot see the dynamic changes leading to these differences. The final way is the FA study of the images. Furthermore, no dynamic effects such as the movement of disclinations or of the fluid in these surface regions are observable. In effect, this surface texture is very stable and the hydrodynamics developed at higher voltage exists above the surface layer (for details see the FA results and discussion).

We prepared additionally a second MBBA cell with a thickness of $12\ \mu\text{m}$ and symmetrical weak anchoring of the liquid crystal. This nematic cell was also subjected to a d.c. voltage with duration of 30 s and amplitude increasing step by step, as described previously. Again we observed the development of flexoelectric domains [33–37] appearing at 2.6 V. The behaviour of the flexoelectric domains in this nematic cell, however was different. The bulk domains were not fixed and could easily move, or glide, in the plane of the figure. The rubbing-induced domains were again attached to the electrodes and could be seen only after focusing the microscope on these regions. We were interested, however, in the cathode region where the flexoelectric domains are formed; this is shown in figure 5. One can also see the interaction of the flexoelectric domain walls with the glass slide producing various kinds of surface disclinations.

The average change of the period of the rubbing-induced domains over the whole photograph is graphically shown in figure 6 for the two cells under study: curve 1 corresponds to the cell with a thickness of $12\ \mu\text{m}$, and curve 2 to the cell with a thickness of $50\ \mu\text{m}$. The curves shown do not depend on the thickness of the cells; furthermore, they appear to show that the variation of the period is inversely proportional to the voltage (the FA results, however, show that this is not the case and the effect is an artefact of the experiment). The change in the periodicity of the rubbing-induced domains began at a voltage of 3.5 V. This experimental fact is in agreement with the electrochemical processes produced in the MBBA layers by the applied d.c. voltage [58–62]. In addition, the behaviour of the flexoelectric domains for the case of MBBA [33–37, 63] clearly show that the electrochemical processes are more active around the

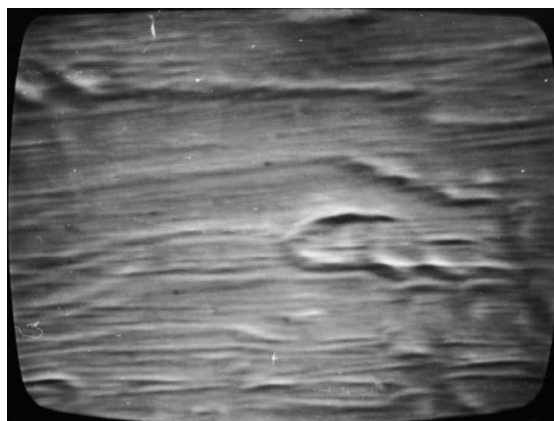


Figure 5. A photograph taken of monitor showing the intersections of the large flexoelectric domains and the rubbing-induced domains in a symmetrically weakly anchored MBBA nematic layer with a thickness of $12\ \mu\text{m}$ at room temperature. The focusing of the polarizing microscope is on the electrode. The applied d.c. voltage has an amplitude of 6.67 V. The polarizer is perpendicular to the domains, and the analyser has been removed. The short side of the photo corresponds to $256\ \mu\text{m}$.

cathode. Taking into account the reduction potential, which is in the range of $-2.4\ \text{V}$ [60, 62] and the ohmic drop of the potential, one obtains a voltage of 3.5 V. Lomax *et al.* [58] described this value as a ‘degradation

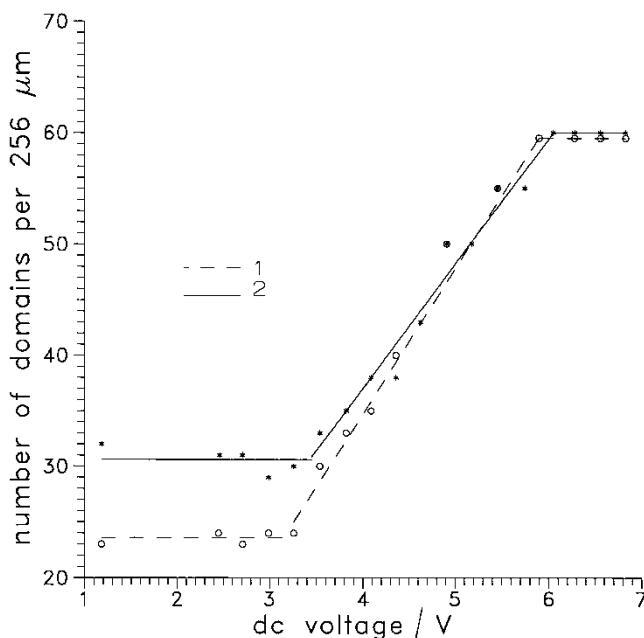


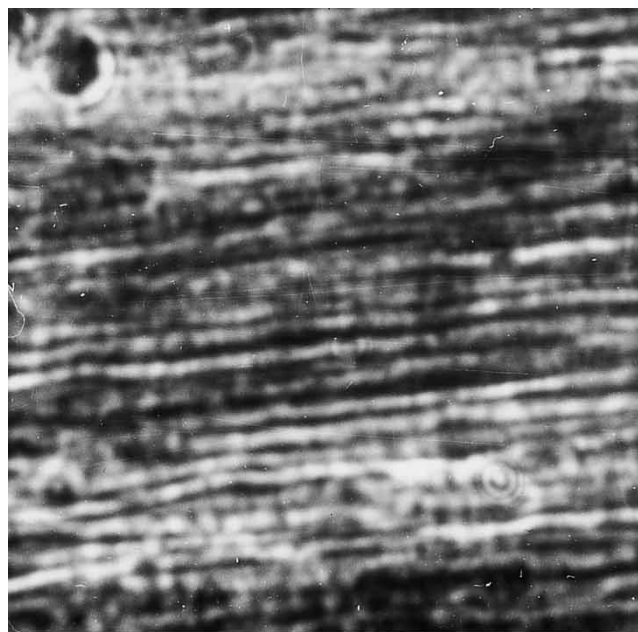
Figure 6. The mean number of rubbing-induced domains per $256\ \mu\text{m}$ (the short side of the photograph) as a function of the applied d.c. voltage. Curve 1 refers to the MBBA nematic layer with a thickness of $12\ \mu\text{m}$ and symmetrical weak anchoring; curve 2 refers to the MBBA nematic layer with a thickness of $50\ \mu\text{m}$ and asymmetrical strong-weak anchoring.

voltage' in the case of MBBA. Voïnov and Dunnet [61] reported a similar voltage (3.8 V) for a MBBA–EBBA mixture. On the other hand, the change in periodicity of the rubbing-induced domains ceased at a voltage of 6 V. Certainly this value is not related to the electrochemical behaviour since the electrochemical processes are also important at higher voltages. This value is probably related to elasto-flexo-dielectric interactions between every pair of adjacent domains.

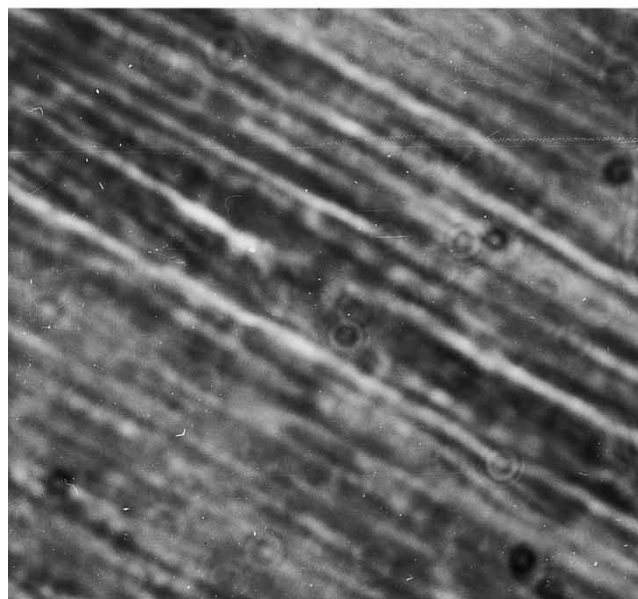
The decrease in period of the rubbing-induced domains is an artefact due to two concurrent effects. First, the initial period of the rubbing-induced domains varies from one place to another and is connected with the local value of the surface tilt of the director and the local value of the surface energy of the liquid crystal; second, as we shall see, the period of the rubbing-induced domains decreases by half, independently of the initial value of the periodicity (it should be noted, however that the greater period decreases by half with a lower voltage in comparison with the smaller periods of the rubbing-induced domains). We shall discuss this problem later.

Furthermore, we studied the behaviour of rubbing-induced domains in a MBBA nematic cell with a thickness of $20\ \mu\text{m}$ under the application of an a.c. electric field with a frequency of 200 kHz, i.e. much higher than the cut-off frequency for charge relaxation. The photomicrographs of figure 7 were obtained using a polarizing microscope with crossed polarizer and analyser. In figure 7(a) one can see the rubbing-induced domains when the direction of the rubbing of the glass plate is along the polarizer; figure 7(b) shows the case when the rubbing direction is along the bisector of the crossed polarizer and analyser. In figure 8(a) one can see the change in surface-induced textures when the cell is exposed to a high voltage of $15\ V_{\text{rms}}$ and a frequency of 200 kHz. Figure 8(b) shows the change when the cell is rotated by 45° . Comparison of the rubbing-induced domains in figures 7 and 8 shows that the surface deformations of the rubbing-induced domains are predominantly θ -polar (or zenithal) and that linear disclinations are present in the domains. This will be discussed in detail in the next section.

Careful study of the rubbing-induced domains under a polarizing microscope shows that they are more visible when oriented along the bisector of the initially crossed polarizers. The existence of dark and bright bands (sometimes red and green in colour) in the rubbing-induced domains clearly shows the existence and variation of the θ -polar (or zenithal) deformations. There are no changes in the domains when the applied a.c. voltage is below $2\ V_{\text{rms}}$. One observes slight changes in the colouring of the picture at approximately $2.5\ V_{\text{rms}}$. The changes continue to increase at a voltage of $3\ V_{\text{rms}}$.



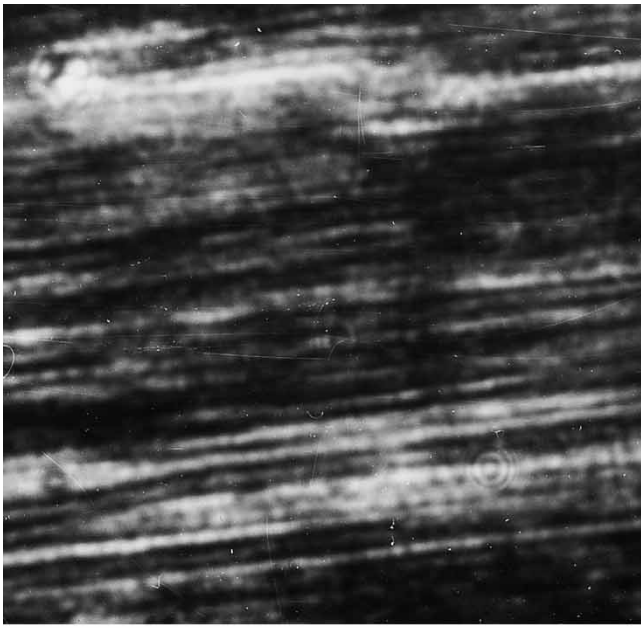
(a)



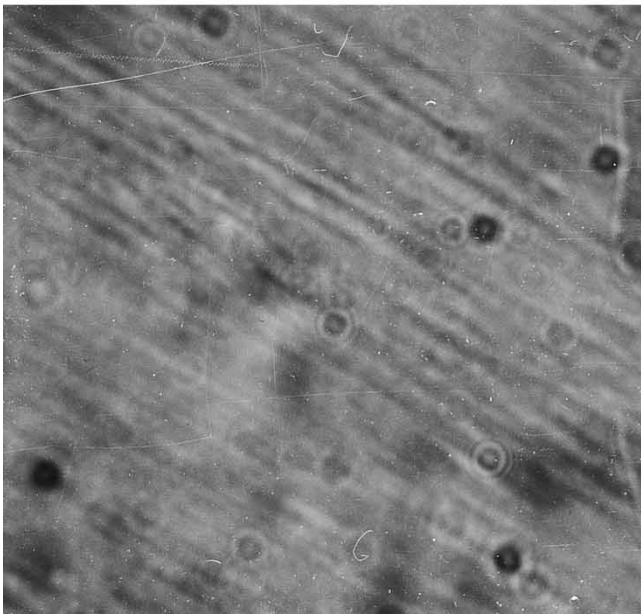
(b)

Figure 7. Photomicrograph of the rubbing-induced domains in a MBBA nematic layer with a thickness of $20\ \mu\text{m}$ and weak anchoring at room temperature; crossed polarizer and analyser. Each side of the photo corresponds to $570\ \mu\text{m}$. The orientation of the rubbing induced domains is: (a) along the analyser; (b) along the bisector of the crossed polarizer and analyser.

The Fréedericksz transition within the domains is visible at $4\ V_{\text{rms}}$ and the director begins to reorient perpendicular to the applied electric field. The periodicity of the rubbing-induced domains, in contrast to the d.c. voltage case, was unchanged. The coloured pictures



(a)



(b)

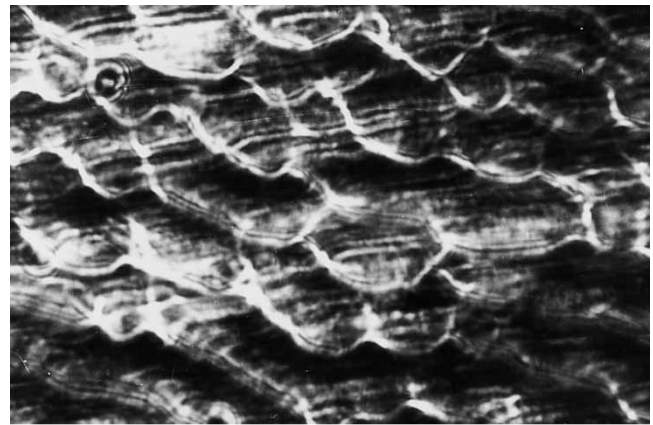
Figure 8. The rubbing-induced domains from figure 7 under an a.c. electric field with a frequency of 200 kHz and an amplitude of $15 V_{\text{rms}}$. The rubbing-induced domains are (a) along the analyser; (b) along the bisector of the crossed polarizer and analyser.

disappear at about $5 V_{\text{rms}}$ followed by the formation of reverse-tilted domains in some areas of the MBBA cell. Further increasing of the a.c. voltage to $15 V_{\text{rms}}$, had no effect.

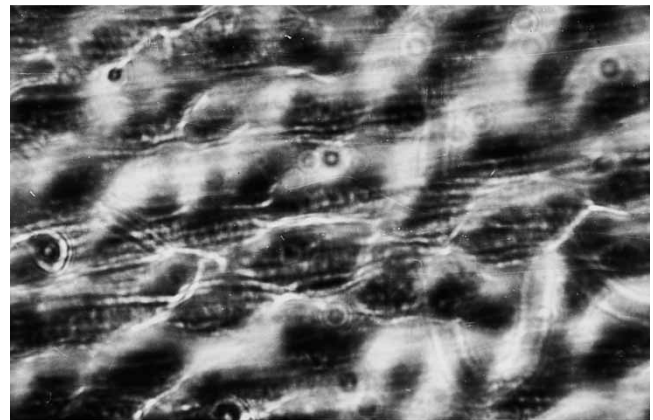
The application of a d.c. voltage across the MBBA nematic layer reveals the existence of disclinations

embedded in the surface domains. At the anode one observes a threshold development of honeycomb injection domains appearing at 5 V, see figure 9 (a). The Orsay Liquid Crystal Group [64] has observed similar domains. It is very important to mention here that at the threshold voltage some of the disclinations embedded in the surface domains jump in the surface region, transforming themselves from rectilinear to complex zig-zag forms which finally form the well known honeycomb domains. At the points of intersection of the disclinations one can see the formation of point disclinations. The details of the deformations, deeply below the anode, can be seen in figure 9 (b).

Additional experiments have been performed with this cell and the rubbing-induced domains around the nematic-isotropic phase transition of the MBBA layer. The surface-induced domains were not clearly visible at



(a)



(b)

Figure 9. Honeycomb injection domains in a MBBA nematic cell of thickness $20 \mu\text{m}$ at room temperature. Crossed polarizer and analyzer, the short side of the micro-photograph corresponds to $570 \mu\text{m}$. The polarizing microscope is focused (a) near the anode and (b) near the cathode.

temperatures slightly below the transition and the voltage only weakly affects their shape. They disappeared completely after the nematic–isotropic phase transition. On decreasing the temperature, the domains reappeared in the nematic phase and the picture was the same. This clearly shows that the rubbing-induced domains are connected with the topography of the confining surface.

3.2. Fourier analysis

The surface-induced deformations and domains observed in this study were very complex. The electro-optical changes in their structure under an applied d.c. voltage were very slow and barely observable. A computer study of some of the observed pictures by Fourier analysis (FA) considerably aids the understanding of what actually happens to these surface-induced structures. As noted already, the most important images were divided into 512×512 pixels. The digitized images are stored and processed on the computer using one-dimensional Fourier analysis. All the FA curves shown are spectral densities (in arbitrary units) plotted against the reciprocal wavelength (in 10^2 cm^{-1}). All these curves can be used for the determination of the space periodicity of the observed surface domains and textures. Finally, we give some FA curves extracted from the general FA results from one arbitrarily preselected line, perpendicular to the rubbing-induced domains. In this way, some dynamic electro-optic effects connected with the study of the MBBA nematic cells can be further elucidated.

The image from figure 2 was digitized and processed by the procedure described. The FA curves for this image are shown in figure 10. It is seen that the liquid crystal only maps the grooves produced by the rubbing of the glass plate (compare curves 1 and 2). Figure 11 shows the FA curves of the initial deformations of the liquid crystal due to the rubbing effect in two semi-free MBBA nematic layers (see figure 3). First, the glass plate was rubbed with a diamond paste spread on a cloth, cleaned rubbed with a soap according to the procedure described, cleaned again with a cloth and then the liquid crystal was deposited onto the glass plate. The thickness of the liquid crystal was a few microns. The orientation and the anchoring of the director at the free surface is a complex problem.† Bouchiat *et al.* [65] have measured for the first time the orientation of the director at the MBBA–air interface and have found that it is inclined at an angle varying

†The authors of this paper thank the *Liquid Crystals* referee for raising the problem of the orientation and anchoring of the director at the MBBA–air interface for a semi-free nematic layer.

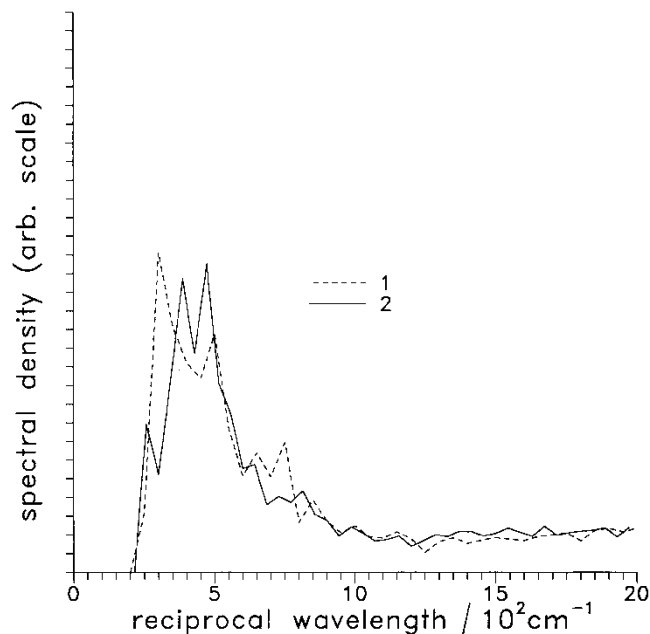


Figure 10. Fourier analysis curves of the rubbing-induced domains shown in figure 2. The dashed curve 1 corresponds to the upper region (containing a liquid crystal) of the photomicrograph; the solid curve 2 corresponds to the lower region of the photomicrograph where liquid crystal is absent.

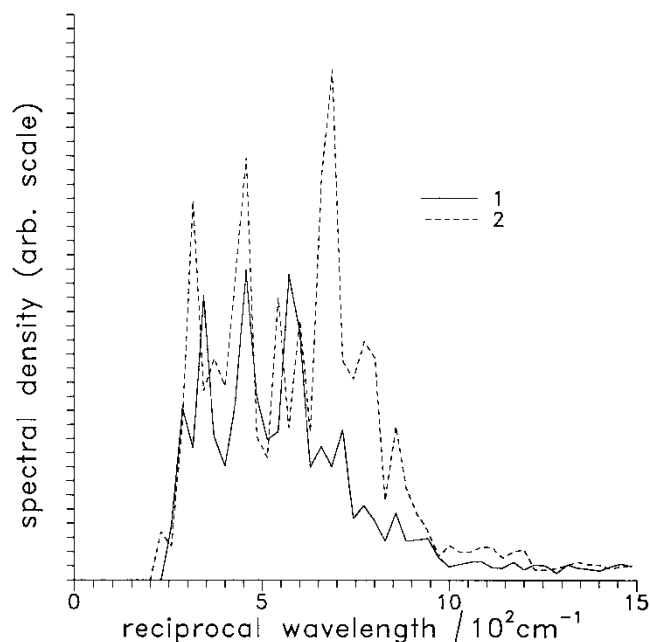


Figure 11. Fourier analysis curves of the rubbing-induced domains shown in figures 3(a) and 3(b). The solid curve 1 corresponds to the photomicrograph shown in figure 3(a); the dashed curve 2 corresponds to that shown in figure 3(b).

with temperature. Furthermore, these authors have proved that the anchoring of the director at the MBBA–air interface is relatively strong. Chiarelli *et al.* [66, 67] have studied this behaviour in detail and have found, first, that far from the nematic–isotropic phase transition the director is tilted with a relatively strong anchoring, $W_s > (0.2–0.4) \text{ erg cm}^{-2}$; second, that near the nematic–isotropic phase transition the director is nearly homeotropic and the anchoring is relatively weak. In addition, the value of the tilt angle and the anchoring can be changed by impurities or other additives. We note that rubbing-induced domains can always be formed in MBBA semi-free layers irrespective of the orientation and anchoring at the liquid crystal–air interface. Although the rubbing was performed manually, the surface domains and the corresponding FA results are almost identical in the two examples (compare figures 3 and 11).

The FA curves, shown in figure 12, were obtained after the computer processing of images extracted from a videotape of the electro-optical behaviour of a MBBA nematic layer, with a thickness of $50 \mu\text{m}$ and asymmetrical strong–weak anchoring (we have designated this cell as **K**). Domains were obtained at the soap-treated electrode serving as cathode where the anchoring of

the liquid crystal is weak. Curve 1 corresponds to the image, designated **K**₀₁, shown in figure 4(a) and was obtained at a voltage of 1.18 V. Curve 2 corresponds to the image, designated **K**₀₈, obtained at a voltage of 4.09 V; a similar image is shown in figure 4(c). Curve 3 corresponds to the image, designated **K**₁₆, obtained at a voltage of 6.28 V; a similar image is shown in figure 4(d). It is important to stress here, that the black stripes, corresponding to the optical images of the large flexoelectric domains, were removed from the pictures since they lead to a large error in the FA curves, especially in the high-frequency region.

The change of the domain periodicity is different in the various regions of the cell and, as noted previously, depends crucially on the local surface energy and the local tilt of the director. In principle, we know *a priori* that the period, at least for the large domains, changes twice on increasing the applied d.c. voltage. Consequently, we decided to compare the FA curve for the **K**₀₁ cell, taken with a double periodicity, with the FA curve for the **K**₀₈ cell (figure 13) as well as the FA curve for the **K**₀₁ cell, taken with a double periodicity, with the FA curve for the **K**₁₆ cell (figure 14). The curves from figure 13 show that the doubling of the greater periods began at a voltage of 3.5 V, while the curves from figure 14 show that the doubling of all the periodic deformations is complete at a d.c. voltage of 6.28 V.

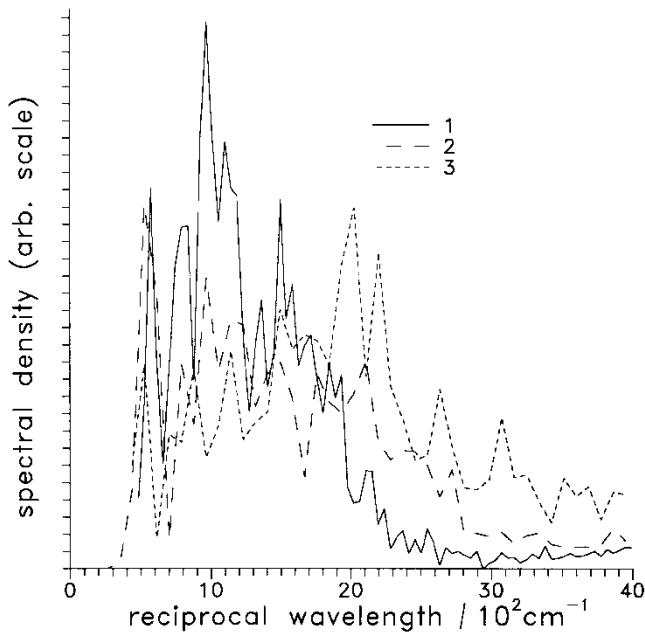


Figure 12. Fourier analysis curves of the rubbing-induced domains shown in figure 4(a) and designated **K**₀₁; rubbing-induced domains designated **K**₀₈, c.f. figure 4(c); rubbing-induced domains designated **K**₁₆, c.f. figure 4(d). The black bands of the large flexoelectric domains are removed from the pictures. Solid line 1: **K**₀₁ at a d.c. voltage of 1.18 V. Dashed curve 2: **K**₀₈ at a d.c. voltage of 4.09 V. Dashed curve 3: **K**₁₆ at a d.c. voltage of 6.28 V.

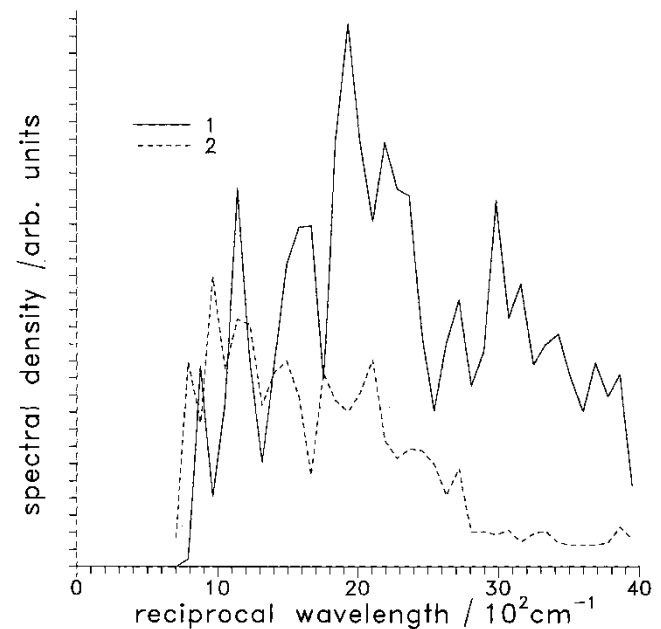


Figure 13. A comparison of FA curves 1 and 2 of the rubbing-induced domains, designated **K**₀₁ with a double periodicity and **K**₀₈ with a real periodicity, respectively. One notes the process of doubling of the domains with the greater periods.

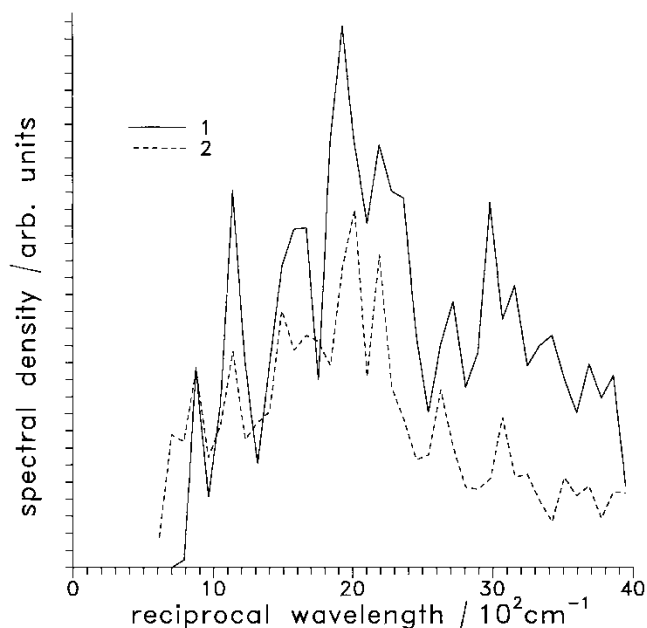
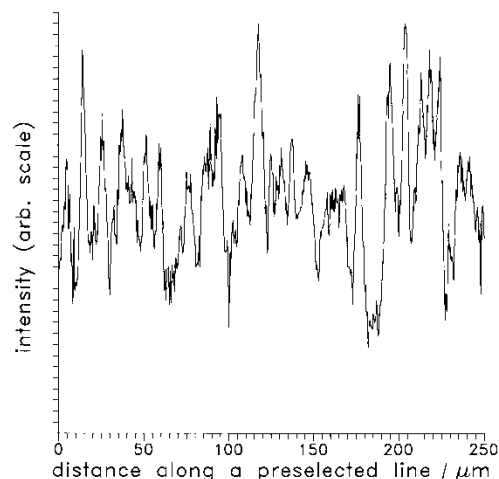


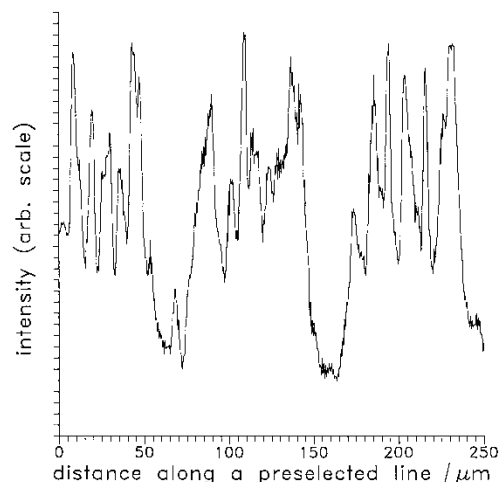
Figure 14. A comparison of FA curves 1 and 2 of the rubbing-induced domains, \mathbf{K}_{01} with a doubled periodicity and \mathbf{K}_{16} with a real periodicity, respectively. One notes the doubling of the period of the rubbing-induced domains over all the wavelengths under observation, and the appearance of domains with new (the smallest) periodicity.

Furthermore, it is evident that new periodic deformations appear at the higher voltage which have the smallest period. Consequently, the linear dependence of the period of the domains as a function of the applied d.c. voltage, as shown in figure 6, is an artefact due to the non-homogeneity of the surface energy and to the different periods of the rubbing-induced domains in the various areas of the cells. It is natural that the larger surface domains are divided with the aid of a smaller d.c. field, while dividing surface domains with smaller periods requires larger d.c. voltages.

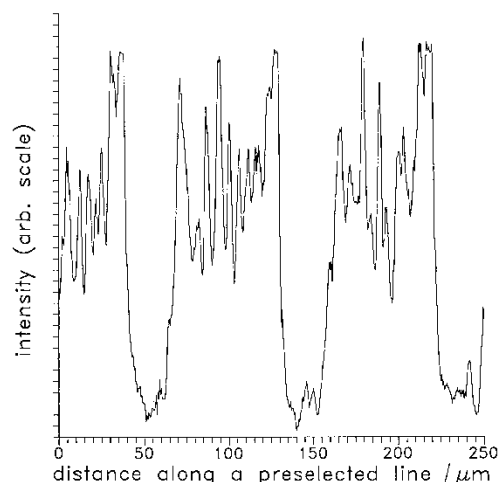
Finally, we present the FA curves along one preselected line from the pictures designated by \mathbf{K}_{01} , \mathbf{K}_{08} and \mathbf{K}_{16} . This line is chosen to be perpendicular to the domains. They are shown in figures 15(a-c). The dynamic changes of similar curves were also studied on the computer monitor. The electro-optical changes were recorded on a video tape and investigated in a dynamic regime. This analysis confirmed the results obtained by the FA of the whole pictures. The large changes in the rubbing-induced domains began at a d.c. voltage of 3.5 V and were complete at 6 V. This experimental study was effective, however, only for low voltages (see the images \mathbf{K}_{01} and \mathbf{K}_{08}). Above 4–5 V we saw very stable domains changing very slowly. Consequently, in the voltage interval above 4–5 V, the usual FA of the whole pictures was more useful than the FA analysis



(a)



(b)



(c)

Figure 15. The Fourier analysis curves along one preselected line which is perpendicular to the domains. (a) The preselected line is from the image \mathbf{K}_{01} , corresponding to figure 4(a); (b) preselected line from image \mathbf{K}_{08} , c.f. figure 4(c); (c) preselected line from image \mathbf{K}_{16} , c.f. figure 4(d).

along one preselected line. The largest changes in the period of the rubbing-induced domains have been observed at 4 V. This voltage coincides with that leading to the Fréedericksz transition. It seems that at this voltage, the division of the domains is most favourable. On the other hand, it should be stressed, as noted previously, that dividing the surface-induced domains with the smaller period requires larger flexoelectric and dielectric energies and really occurs at higher voltages.

4. Discussion

As noted previously, a number of authors [3, 7, 9, 43–49] have observed similar surface-induced domains. In addition, the division of the surface-induced domains under the application of a d.c. voltage described in this paper, and that of the flexoelectric domains observed by Greubel and Wolff [68] are very similar. We have observed also that the period of the domains decreases by about half when the focusing of the microscope is outside the surface-induced domains, deep in the liquid crystal. This optical behaviour of the domains has been explained by Aero [69]. When the microscope is focused within the domains and the orientation of the liquid crystal on the grooved surface is homeotropic [69] or highly-tilted (this paper), one can see the valleys and peaks of the grooved surface. When the focusing is outside the domains, deep in the liquid crystal, the valleys and peaks are not seen and the periodicity of the domains is halved. Furthermore, the experimental results clearly show that the dark regions of the rubbing-induced domains determine the liquid crystal orientation deep in the cell (the polarizer is perpendicular to the domains, and the analyzer has been removed).

The bright regions of the domains are connected with line defects existing in their structure [70]. Such lines disturb the homogeneity of liquid crystal displays, as has been noted by Kimura *et al* [71]. It has been well known for a long time that the switching of a homeotropic (in our case highly-tilted) orientation to the planar one is accompanied by the appearance of line disclinations, as first observed by Saupe [72]. On the other hand, the irregular topography of the treated surface, combined with the rubbing of 1 μm diamond grains, often leads to the observation of many bright bumps or spots [73] (see figure 3 here and figure 2 in [73]) in which the liquid crystal orientation is conically degenerate. The existence of point disclinations [70] in their structure is questionable. We only know that point disclinations usually appear in combination with, for example, linear disclinations, and holes [74–77]. In contrast, bright bumps or spots have often been observed in the electro-hydrodynamics of liquid crystals

[78, 79] and are discussed in connection with the homogeneity of liquid crystal devices [80].

In order to propose a model for the organization of the domains we must know the order of magnitude of the surface anchoring of the liquid crystal and should invoke flexoelectric effects in order to explain the division of the domains under a d.c. voltage. The azimuthal anchoring of the MBBA liquid crystal, confined between two soap-treated glass plates covered with a semitransparent conductive layer, is very weak as revealed by the strong thermal fluctuations of the director and by the electro-optical behaviour of such layers including, for example, the Fréedericksz transition, the appearance of the flexoelectric domains and some electro-optical effects in large pitch cholesterics [33–42]. This energy should be in the range of 10^{-3} – 10^{-4} erg cm^{-2} . The θ -polar, or zenithal, anchoring energy should be of the same order of magnitude since the alignment of the nematic layers is usually tilted. This surface energy can be estimated from the equation of de Gennes [81] $W_{S\theta} = (K/2b)$, where K is the mean elastic constant of the liquid crystal and b is the thickness of the surface disclination, which for our experiment was measured to be around 2.5 μm (θ -surface disclinations can often be observed in one semi-free MBBA nematic layer when the liquid crystal is covering one soap-treated glass plate). The estimate of the θ -polar surface energy for this particular case is 2×10^{-3} erg cm^{-2} .

Another very important question relates to the value of the period of the rubbing-induced domains. It should correlate with the size of the diamond grains, i.e. around 1 μm . Their size was observed under a polarizing microscope [82–86]. Our experimental results, however, clearly show that the real periodicity of the liquid crystal orientation is much lower (compare 1 μm versus 10–20 μm , see the experimental results). In our opinion, the periodicity of the rubbing-induced domains is determined by the deeper grooves where linear disclinations appear. This can also be seen from the kind of the domains shown in figure 2. Another cause, leading to the lower periodicity of the grooving, is the statistical distribution of the diamond grains during the rubbing process [82]. The remaining grooves have much smaller amplitudes and higher periodicity, and the second rubbing with the soap covers them completely.

Our model with which to explain the surface-induced domains and their behaviour in applied d.c. or a.c. voltages must encompass the following points:

- (a) The rubbing produces a nearly sinusoidal grooving of the surface.
- (b) The deeper grooves determine the appearance of linear disclinations and are at a distance between 5 and 10 μm .

- (c) The existence of a high tilt of the director, oriented along the grooves.
- (d) The existence of relatively weak θ , ϑ polar-azimuthal anchoring of the liquid crystal.
- (e) The creation of flexoelectric deformations under the application of a d.c. voltage due to the non-homogeneity of the electric field.

The model is shown schematically in figure 16. We suppose that the director lines are closed in the valleys containing predominantly bend deformations. We cannot exclude, however, the existence of splay-bend deformations transforming the circles into more elongated figures. It is important to note that during the experiment we noticed no twist deformations. In the centre, where the bend or splay-bend deformations are greatest, there should exist surface disclinations (in some cases, for example strong anchoring, this surface disclination could be replaced with the disclination of strength $s = +1/2$ [72]). The liquid crystal orientation in the valleys should match the orientation of the rest of the liquid crystal contained along the grooves. This matching is achieved by the creation of a number of $-1/2$ wedge disclinations, which should exist above the valleys. On the other hand, above the peaks, the liquid crystal is oriented along the grooves, figure 16(a). Under the polarizing microscope and by illumination with light polarized perpendicular to the direction of the domains, the regions of the liquid crystal above the peaks appear dark while those in the valleys are bright.

This model is a slight variation of that proposed by Saupe [72] for matching the homeotropic orientation of the liquid crystal at the boundary with a planar orientation below the boundary. The highly energetic $+1/2$ wedge disclinations in the Saupe model are replaced by the less energetic surface disclinations in our model due to the weak surface anchoring [87–90]. Similar models to that proposed by Saupe can be found in the works of Cheng [91] and Cheng *et al.* [92].

In an applied high frequency a.c. electric field, the $-1/2$ wedge disclinations cannot be eliminated and the liquid crystal cannot be oriented homogeneously along one direction. Under a d.c. voltage, however, various flexoelectric effects come into play. We accept that the flexoelectric deformations appear first just above the valleys, figure 16(b), due to the non-homogeneity of the electric field caused by the grooving of the surface and charge injection from the cathode. Further increase in the d.c. voltage causes the increase of these unstable deformations and at some moment one observes an abrupt doubling of the domains as shown in figure 16(c). These flexoelectric deformations require the creation of a new surface disclination and a new $-1/2$ wedge singularity line.

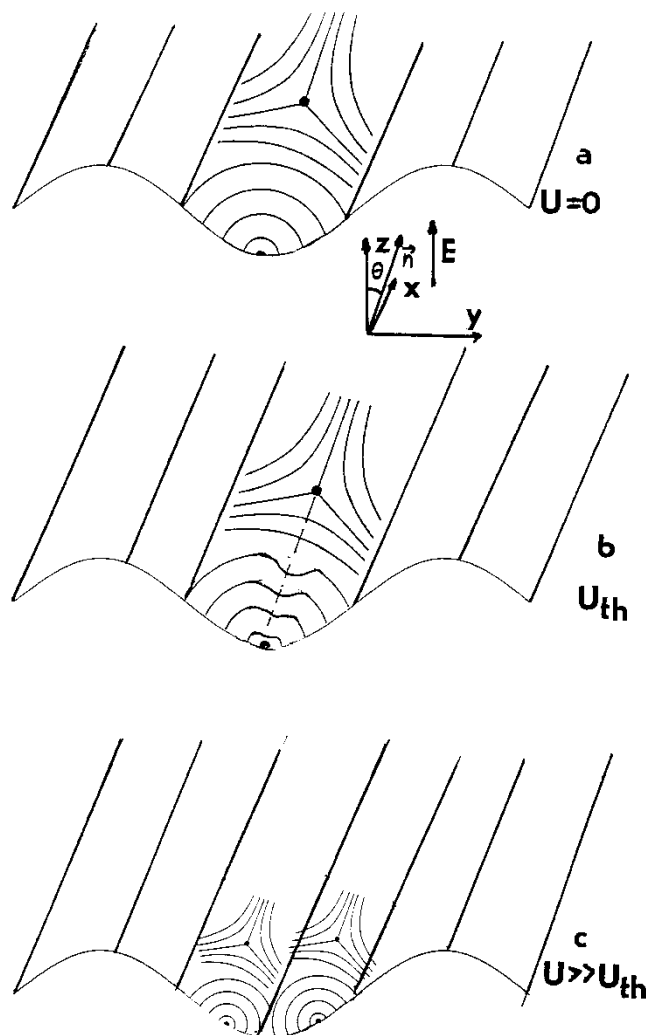


Figure 16. A schematic model representing the doubling of the initial rubbing-induced domains. (a) Initial view of the rubbing-induced domains incorporating in the structure a surface disclination and a wedge $-1/2$ linear disclination; (b) beginning of the doubling of the domains under the application of a d.c. voltage; (c) final doubling of the domains under the application of a d.c. voltage of higher amplitude.

Evidently in the final picture all the known flexoelectric torques come into play: first, the gradient flexoelectric torques connected with the non-homogeneity of the electric field due to the rubbing effects and the accumulation of injected charges, etc. [93, 94]; second, the bulk flexoelectric torques, first considered by Meyer [95]; finally, the flexoelectric surface torques, connected with the weak anchoring of the liquid crystal and with the so-called polar flexoelectric effect [96, 97]. The detailed calculation of these deformations should include the elastic, dielectric and flexoelectric energies of the liquid crystal as well as the flexoelectric-elastic energy of the surface and bulk disclinations. Here we note that

the existence of space charges near the cathode, sustained by the current under d.c. voltage excitation, is in agreement with the observation of bulk flexoelectric domains (also called flexo-dielectric walls) near the cathode.

5. Summary

In conclusion, we have observed experimentally surface irregularities (rubbing-induced domains, bumps, etc.) in MBBA nematic cells due to the special treatment of the electrodes. We have used various techniques such as polarizing microscopy, the shadowgraph technique and computer processing of the layer parts of the images obtained in the experiment. Our study is illustrated with many photomicrographs, some of them divided into 512×512 pixels and processed on a computer with the aid of optical Fourier analysis. Our observations, and in particular the Fourier Analysis curves, clearly show that these rubbing-induced domains divide under the application of a d.c. voltage due to complex flexoelectric effects which begin at 3.5 V and finish at 6 V. Further, we have observed that the well known flexoelectric domains studied in several papers [33–37, 63] are situated near the cathode and that they disturb the rubbing-induced domains either where the bulk flexoelectric domains meet the electrode (this is the case, when the rubbing-induced domains have a smaller period) or in the whole region (when the rubbing-induced domains have a larger period) [63].

The application of an a.c. voltage with a sufficiently high frequency above the cut-off for charge relaxation, aids our understanding of the structure of the rubbing-induced domains, including surface and bulk disclinations. We propose a model, similar to that originally advanced by Saupe [72] which, however, accounts for the weak anchoring of the liquid crystal on a nearly sinusoidal surface, and for the existence of initial tilt. This model is further elaborated to include the flexoelectric phenomenon which leads to a doubling of the periodicity of the rubbing-induced domains, independently of the magnitude of their period.

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