

## Influence of anchoring strength on focal conic domains in smectic films

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We present a study of focal conic domains in smectic-*A* liquid-crystal films on solid substrates. The antagonistic anchoring conditions of the film surfaces, random planar at the substrate interface and homeotropic at the air interface, enforce the formation of focal conic domains the lateral size of which is dependent on the film thickness. The strength of the planar anchoring on the solid substrate is systematically varied by coating the substrate with special alkoxy silane compounds. For each anchoring strength value, the relation between the size of the focal conic domains and the film thickness is determined. Increasing the planar anchoring strength influences the size-thickness relation and leads to the formation of larger focal conic domains.

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### I. INTRODUCTION

Defect structures in liquid crystals (LCs) are of interest from both fundamental and applied viewpoints. For instance, the distortion of the director field  $\vec{n}(\vec{r})$  ( $\vec{n}$  designating the preferred direction of the long axis of the rodlike molecules) in a nematic phase by small water droplets or solid particles leads to special colloidal interactions [1], which may be used for the design of self-assembling microsystems [2–4].

In smectic LCs, defect structures are characterized by the constraint of maintaining a constant smectic layer thickness. This constraint results in the formation of focal conic domains (FCDs) [5,6] in which the smectic layers are wrapped around two defect lines, an ellipse and a hyperbola, which are localized in two perpendicular planes and pass through each other's focal point. In  $\mu\text{m}$ -thick films, FCDs can be generated on purpose and often self-organize in a regular two-dimensional lattice [7–9]. In recent years, several studies explored methods of controlling the size and the arrangement of FCDs in such films—e.g., by confining FCDs into microchannels [10–12] or anchoring patterns [13,14] on the substrate. It was also shown that FCDs may serve as matrices for the self-assembly of soft microsystems [12].

The formation of FCDs is the response of the smectic LC to a frustration between an external influence inducing a bent configuration and the incompressibility of the smectic layers. For instance, in a cell with homeotropic anchoring ( $\vec{n}$  is oriented perpendicularly to the interface) at both interfaces, the smectic layers usually form a simple stack with the layer planes aligned parallel to the interfaces; if an electric field of sufficient strength is applied perpendicular to the layers and the LC possesses a negative dielectric anisotropy, the formation of FCDs is induced [15]. FCDs also form in hybrid cells which possess homeotropic anchoring at one interface and planar anchoring ( $\vec{n}$  being parallel to the interface) at the second interface. If the planar aligning interface is rubbed so that a preferred in-plane orientation of  $\vec{n}$  exists, fragmented FCDs are generated [16,17].

Instead of using a cell, FCDs are easily generated by preparing a smectic film on a planar anchoring substrate. The

free surface (interface to air) of the film usually exhibits a strong homeotropic anchoring. If the planar anchoring substrate imposes a preferred in-plane orientation of  $\vec{n}$ , such as surfaces of crystals like mica or  $\text{MoS}_2$ , linear defect structures are generated [18]. If no preferred in-plane direction on the substrate surface exists—e.g., on a liquid or a solid coated with a suitable polymer—toric FCDs are obtained in which the singular lines (ellipse and hyperbola) adopt the shape of a circle and a straight line [7–9]. Thus, it is obvious that the anchoring of the smectic LC at the substrate interface is a key issue for the generation and control of FCDs in such films. It was recently shown [13,14] that FCDs can be confined to certain regions on the substrate by patterning the substrate with different anchoring *types*. On the other hand, there exists no systematic study of the influence of the anchoring *strength* on the structure of FCDs in smectic LC films. In a number of studies [15,19–23], however, the generation of FCDs or similar structures was used to obtain a quantitative estimation of the anchoring strength of smectic LCs on various substrates. A comprehensive theoretical treatment of the energy related with the formation of FCDs is given in [24].

Here, we present a study of FCDs on different substrates possessing different planar anchoring strength values. We show that the relation between the thickness of the smectic film and the size of the FCDs is essentially determined by the strength of the random planar anchoring on the substrate.

The anchoring strength is usually defined by means of the energy which is related to the difference between the actual direction of  $\vec{n}$  and the direction preferred by the interface (often designated as easy axis) [25]. Whereas for nematic LCs oblique anchoring states, with  $\vec{n}$  being oriented at some arbitrary angle at the interface, are frequently observed, for smectic-*A* LCs homeotropic and planar anchorings are by far the most important configurations. In the present study, we consider substrates which favor a planar anchoring of a smectic-*A* phase. However, external constraints—for instance, the vicinity of another interface possessing a strong homeotropic anchoring condition—may enforce a homeotropic anchoring also on these substrates. The energy difference  $\Delta F$  between the two anchoring states on the substrate is given by

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$$\Delta F = -\Delta\sigma_{sub}A, \quad (1)$$

where  $A$  is the substrate area and  $\Delta\sigma_{sub} = \sigma_{\parallel} - \sigma_{\perp}$  the surface energy difference between planar and homeotropic anchorings on the substrate. For the purpose of the present study and in accordance with a theoretical model [7] which we use below to interpret our results, we take the magnitude of  $\Delta\sigma_{sub}$  as a quantitative measure of the planar anchoring strength of our substrates.

## II. EXPERIMENT

As substrates, we used silicon wafers; the anchoring conditions can be controlled by treating the substrate surface with aqueous solutions containing the alkoxy silane compounds MAP (*N*-methyl-3-aminopropyltrimethoxysilane) and DMOAP (*N,N*-dimethyl-*N*-octadecyl-3- amino propyl trimethoxy-silyl chloride) [26]. Coating a silicon substrate with MAP results in random planar anchoring, whereas coating with DMOAP leads to homeotropic anchoring conditions because the DMOAP molecule possesses a side chain extending away from the substrate surface [26]. Thus, coating a silicon substrate with a mixed solution, containing a large amount of MAP and a small amount of DMOAP, produces also a substrate possessing a random planar anchoring, but the anchoring strength is slightly smaller compared to that of a substrate treated with a pure MAP solution. With increasing concentration of DMOAP in the solution, the planar anchoring strength of the substrate will be further reduced until it becomes zero and a transition to homeotropic anchoring takes place; as will be explained below, this anchoring transition is induced already by a very small addition of DMOAP to MAP (at a mole fraction  $x_{DMOAP}$  in the silane mixture of the order of  $5 \times 10^{-4}$ ). In this way, the planar anchoring strength of the substrate can be continuously varied between zero and the value obtained with a pure MAP solution.

MAP and DMOAP were obtained from ABCR Company, Kalsruhe, Germany. Diluted aqueous solutions of both silane compounds were prepared and appropriate amounts of these solutions were mixed in order to obtain values of  $x_{DMOAP}$  ranging from 0 (pure MAP) to  $10^{-3}$ . The prepared mixtures were further diluted with water until the overall concentration of silane in water corresponds to  $3 \times 10^{-3}$  mol/l. Before treating a silicon wafer with a silane solution, the wafer is cleaned with piranha solution and subsequently rinsed thoroughly with millipore water. The cleaned silicon wafer is immersed for 5 min in the silane solution, washed with pure water, dried, and then cured for 1 h at 110 °C, resulting in a chemisorbed silane layer permanently bonded to the substrate [26].

As LCs, we used compounds of the *n*CB series (4-cyano-4'-alkylbiphenyl, *n* designating the number of carbon atoms in the alkyl chain) obtained from Synthon Chemicals, Wolfen, Germany. We studied 8CB, 9CB, and a mixture of 75% 8CB with 25% 6CB (corresponding to an effective chain length of 7.5). All LCs show the phase sequence smectic-*A*-nematic-isotropic with increasing temperature. LC

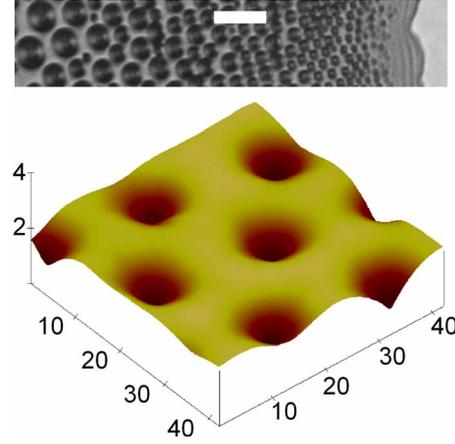


FIG. 1. (Color online) Top: optical micrograph of a flat smectic droplet demonstrating the relation between FCD size and LC film thickness. On the right side, the edge of the droplet and the bare substrate is seen; the local thickness of the LC film increases from right to left. The scale bar corresponds to 10  $\mu\text{m}$ . Bottom: atomic force microscope (AFM) image of the surface of a smectic film containing FCDs; each FCD causes a depression in the air interface [9,14]. The numbers describe the  $x$ ,  $y$ , and  $z$  coordinates in  $\mu\text{m}$ ; the depth of the surface depressions is of the order 1  $\mu\text{m}$ .

films on the substrates were produced by placing at room temperature a tiny amount of LC on the substrate. The temperature was raised to the range of the nematic phase where the LCs formed flat droplets which reached a quasistatic shape after a few minutes. The samples were then slowly cooled across the nematic to smectic-*A* transition where the FCDs formed. Thus, our LC films are in fact flat droplets, the thickness of which increases from the edge to the center; near the edge, the thickness increases typically by a value between 50 and 100 nm per each  $\mu\text{m}$  distance from the edge, this slope decreasing toward the central region of the droplet.

Figure 1 shows an optical micrograph of FCDs in the region near the edge of a smectic droplet. Directly at the edge (which is seen at the right margin of the micrograph) and until a distance of a few  $\mu\text{m}$ , FCDs are not present; then, tiny FCDs appear, the size of which increases with increasing distance from the edge (corresponding to increasing values of the local film thickness). In the thicker regions, where the diameter of the FCDs amounts to several  $\mu\text{m}$ , a certain polydispersity in size is observed as the space between large FCDs is sometimes filled with small FCDs.

We use atomic force microscopy (AFM) measurements for the quantitative determination of the size of the generated FCDs. Each FCD induces a cone-shaped depression in the LC-air interface which can be imaged by AFM [9] (cf. Fig. 1). The diameter  $2r$  of the FCDs is obtained from the distance between the centers of two neighboring and equally sized FCDs. The AFM data also allow the determination of the local thickness of the LC film on the substrate: The AFM images of the film surface were recorded in a way that a small portion of the bare substrate (without the LC film) was also contained in the image. The local film thickness is then obtained from the difference to the  $z$  data of the bare substrate. Experimental details of the AFM measurements can be found in [14].

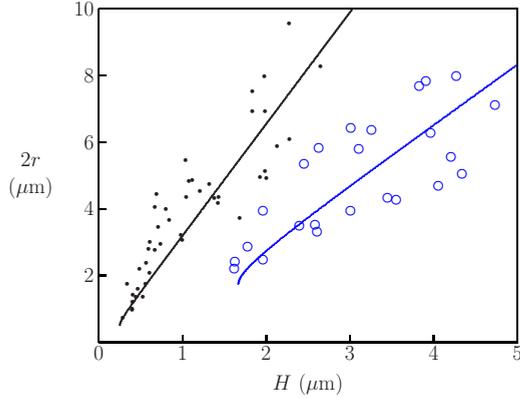


FIG. 2. (Color online) Dependence of FCD diameter  $2r$  on the local thickness  $H$  of smectic LC films (compound 8CB) on two substrates coated with different mixed MAP and DMOAP layers. Small dots:  $x_{DMOAP}=0.7 \times 10^{-4}$ . Open symbols:  $x_{DMOAP}=4.0 \times 10^{-4}$  (the values of  $x_{DMOAP}$  refer to the mole fraction of DMOAP in the silane mixture without solvent). Solid lines are fits according to Eq. (3).

### III. RESULTS AND DISCUSSION

Figure 2 shows experimentally determined values of the diameter  $2r$  as a function of the local thickness  $H$  of the LC film on two different substrates coated with silane layers possessing different values of the planar anchoring strength. In both cases, a certain film thickness value  $H_c$  is observed below which FCDs are not formed. In this region of very thin film thickness  $H < H_c$ , the strong homeotropic anchoring of the air interface enforces homeotropic anchoring also at the substrate interface, resulting in a defect-free film consisting of plane parallel smectic layers (the energy gain of a planar anchoring at the substrate would be smaller than the energy cost of creating FCDs). As to be expected, the smaller the amount of DMOAP in the substrate coating (i.e., the larger the planar anchoring strength of the substrate), the smaller the value of  $H_c$ . In the thickness range above  $H_c$ , a linear relation (with the exception of the immediate vicinity of  $H_c$ ) between the size  $2r$  of the FCDs and the thickness  $H$  of the LC film is observed; the slope of this linear  $2r$  vs  $H$  curve decreases with increasing amount of DMOAP in the substrate coating (i.e., with decreasing planar anchoring strength of the substrate). Thus, our experimental observations concerning the influence of the planar anchoring strength can be qualitatively summarized as follows: with increasing anchoring strength, the size of the generated FCDs increases for a given film thickness, the growth of the FCDs with increasing film thickness becomes more pronounced, and the minimum film thickness required for the generation of FCDs decreases.

The FCDs formed on the MAP- and DMOAP-coated substrate possess a circular shape. This observation is of relevance with respect to a recent study [27] in which the surface pretilt angle of the easy axis was varied with a technique that is similar to our method of varying the anchoring strength: in [27], the substrate was coated with a mixture of two polymers, one of which possesses a side chain favoring homeotropic anchoring. The concentration of the side chains

in the final coating was dependent on the curing temperature, and the pretilt angle increased with decreasing side chain concentration from 0 to  $\approx 80^\circ$ . A pretilt angle considerably smaller than  $90^\circ$  is expected to influence the shape of the FCDs [15]. There may be several reasons why we do not observe this behavior: In [27], the surfaces were rubbed after coating in order to induce a homogeneous azimuthal direction of the pretilt; in our case, the surfaces were not rubbed and there could be a random distribution of pretilt directions which cancel each other. Another reason might be that our measurements were not done in the relevant range of anchoring strength values, since changes of the pretilt angle can be expected mainly in the region where  $\Delta\sigma_{sub} \approx 0$ . However, the most important difference between [27] and our study is probably that we study a smectic instead of a nematic phase. For a smectic-A phase, any surface pretilt value different from 0 or  $90^\circ$  is unfavorable because it would require a “melting” of the smectic layer structure at the surface [24]. Thus, for a smectic-A phase the most favorable orientation of the easy axis could be still at pretilt values close to  $90^\circ$  even if a nematic phase on our substrate would show a different pretilt angle.

We now turn to a more quantitative discussion of our results. The relation between the film thickness and the diameter of FCDs has been considered by Fournier *et al.* [7] who studied smectic films possessing an air interface and floating on the isotropic phase of the same material (the smectic-isotropic interface exhibits random planar anchoring conditions similar to the smectic-substrate interface of our case). The energy  $F$  of a FCD in such a film can be written as [7]

$$F = 2\pi\beta KH + 2\pi\alpha Kr - \Delta\sigma_{sub}\pi r^2 + \frac{\pi\sigma_{air}}{12H^2}r^4. \quad (2)$$

The first two terms take the energy of the two singular lines and the curvature energy of the FCD into account;  $K$  is a mean elastic constant, and  $\alpha$  and  $\beta$  are—within the framework of [7]—dimensionless unknown constants (a detailed theoretical description of the curvature energy of a FCD has been given in [24]). The third term is the same as in Eq. (1) (energy gain of planar anchoring), and the last term describes the energy cost of the increased surface area due to the deformation of the air interface induced by the underlying FCD ( $\sigma_{air}$  being the surface tension of the smectic-A–air interface). Equation (2) predicts a minimum of  $F$  with respect to  $r$  provided that  $r > r_c = 3\alpha K / (2\Delta\sigma_{sub})$  [7], and a nearly linear relationship between film thickness  $H$  and radius  $r$  is obtained:

$$H = \left[ \frac{\sigma_{air}r^3}{6\Delta\sigma_{sub}\left(r - \frac{\alpha K}{\Delta\sigma_{sub}}\right)} \right]^{1/2}. \quad (3)$$

For  $r \gg \frac{\alpha K}{\Delta\sigma_{sub}}$ , Eq. (3) can be rewritten as

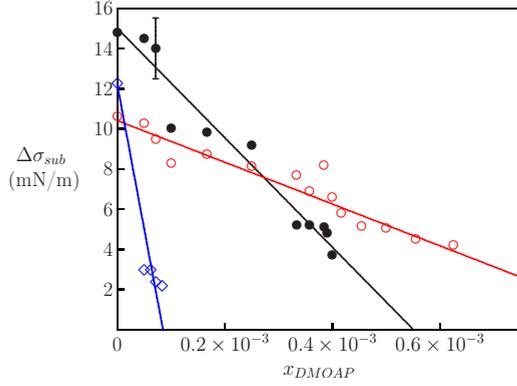


FIG. 3. (Color online) Dependence of planar anchoring strength  $\Delta\sigma_{sub}$  on the mole fraction of DMOAP in the substrate coating for the LC compounds 8CB (●), 9CB (○), and 6CB-8CB mixture corresponding to 7.5CB (◇). The error is of the order of  $\pm 1.5$  mN/m; a typical error bar is shown for one 8CB data point.

$$r = \left( \frac{6\Delta\sigma_{sub}}{\sigma_{air}} \right)^{1/2} H. \quad (4)$$

Thus, the slope of the linear part of the  $r$  vs  $H$  curves shown in Fig. 2 should be proportional to  $\sqrt{\Delta\sigma_{sub}}$ , which fits well to our expectation that the planar anchoring strength of our substrates decreases with increasing amount of DMOAP in the substrate coating.

The value  $r_c = 3\alpha K / (2\Delta\sigma_{sub})$  represents in the model of Eq. (2) the lower limit of the FCD radius. The corresponding value of the film thickness  $H(r_c)$  corresponds to our experimentally observed value  $H_c$  below which FCDs do not form. In order to fit our experimental  $2r$  vs  $H$  data (exemplified in Fig. 2) by Eq. (3), we can obtain from the experimental value of the minimal FCD diameter the ratio  $\alpha K / \Delta\sigma_{sub}$  and from the slope of the  $2r$  vs  $H$  curve the value of  $\Delta\sigma_{sub}$ . We set  $\sigma_{air}$  to 30 mN/m [28] and applied the constraint that the value of  $\alpha K$  should be constant for a given LC compound. The solid lines in Fig. 2 are two examples of calculated [using Eq. (3)] curves fitted to the experimental results.

The values of  $\alpha K$ , obtained for the three LCs under investigation, are  $1.1 \times 10^{-9}$  N (7.5CB),  $1.7 \times 10^{-9}$  N (8CB), and  $2.4 \times 10^{-9}$  N (9CB), indicating that the curvature energy of the FCDs increases with increasing alkyl chain length of the LC molecules. The values of  $\Delta\sigma_{sub}$ , obtained from the fitting process, are shown in Fig. 3 as a function of the composition of the substrate coating for the three LC systems studied here. The three LCs behave qualitatively similar, but there are clear quantitative differences: For the mixture corresponding to 7.5CB,  $\Delta\sigma_{sub}$  decreases rapidly even for very small amounts of DMOAP in the silane mixture and it is difficult to make a definite conclusion on the  $\Delta\sigma_{sub}$  vs  $x_{DMOAP}$  relation. For 8CB and 9CB, however, a linear decrease of  $\Delta\sigma_{sub}$  with increasing DMOAP content  $x_{DMOAP}$  in the substrate coating is observed. Assuming a linear relation for all three LCs and extrapolating to  $\Delta\sigma_{sub} = 0$ , we can determine a value  $x_{DMOAP}^0$  corresponding to a neutral substrate favoring neither planar nor homeotropic anchoring; for  $x_{DMOAP} > x_{DMOAP}^0$ , the substrate would favor a homeotropic

anchoring. For the three LCs under investigation, the following values of  $x_{DMOAP}^0$  are obtained: 7.5CB,  $0.8 \times 10^{-4}$ ; 8CB,  $5.5 \times 10^{-4}$ ; and 9CB,  $10.5 \times 10^{-4}$ . The data indicate that with increasing alkyl chain length a planar anchoring of the smectic-A phase on the MAP- and DMOAP-coated substrates is favored. This is in contrast to the behavior of the nematic phase of the  $n$ CB compounds which have been studied on gold substrates coated with self-assembled thiol monolayers. In that case, it was observed that increasing the alkyl chain length favors a homeotropic anchoring of the nematic phase [29]. However, we should note that the two studies are not directly comparable: In [29], the anchoring behavior at a single interface of thick (several tens of  $\mu$ m) LC films is probed, whereas the results of the present study are based on a structure formation process involving both interfaces (and the complete film) of considerably thinner LC films.

The values of  $\Delta\sigma_{sub}$  determined in the present study are in the range between 0.002 and 0.015 N/m. In previous studies, anchoring strength values have been obtained which vary over a wide range. For smectic-A–liquid interfaces (as liquid phase glycerine-lectithin mixtures, pure glycerin, water, or the isotropic phase of the LC was used), values between  $3 \times 10^{-7}$  and  $10^{-4}$  N/m were observed [7,19,21,22]. At interfaces to solid substrates (SiO- or lectithin-coated glass) values between  $4 \times 10^{-5}$  and 0.01 N/m have been determined [15,20]. The so far largest values were obtained for 8CB on MoS<sub>2</sub>, where  $\Delta\sigma_{sub}$  amounts between 0.0032 and 0.02 N/m [23]. The value 0.015 N/m, which we have determined for 8CB on a silicon wafer coated with pure MAP, is in the same range and confirms the general tendency that solid substrates may possess somewhat higher anchoring strength values compared to liquid substrates.

#### IV. CONCLUSION

We have studied FCDs in smectic-A LC films on solid substrates imposing random planar anchoring on the LC material. The strength of the planar anchoring is systematically varied by coating the substrates with silane layers consisting of the two silane compounds MAP and DMOAP which produce, in pure form, planar or homeotropic anchoring, respectively. For each substrate, the relation between size  $2r$  of the FCDs and thickness  $H$  of the smectic film is determined by AFM measurements. Increasing the planar anchoring strength results in the formation of larger FCDs for a given value of  $H$  and in a larger slope of the  $2r$  vs  $H$  curve. Our results are well described by a model of Fournier *et al.* [7], in which the anchoring strength of the substrate is described by the difference  $\Delta\sigma_{sub}$  between the surface energies of planar and homeotropic anchoring of the smectic-A phase on the substrate. We find that the magnitude of  $\Delta\sigma_{sub}$  decreases linearly with increasing amount of DMOAP in the substrate coating. Comparing the three LC materials under investigation, our results indicate that for compounds with longer

alkyl chains a larger amount of DMOAP in the substrate coating is necessary to induce a change from planar to homeotropic anchoring. The study of the relation between the FCD diameter and the film thickness offers also a method for the quantitative determination of the planar anchoring strength of a smectic-A phase on a solid substrate.

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