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A STRUCTURAL STUDY OF TEMPERATURE INFLUENCE ON THE EFFECT OF GRATED SUBSTRATES ON SMECTIC LIQUID CRYSTAL FILMS

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Abstract We present a preliminary analysis of the results of our structural measurements on the influence of temperature variation on the orientation of smectic liquid crystal films deposited on photolithographed gratings on glass gratings. These gratings have periods of 10μm with depths ranging up to 2 - 3μm. We previously found that both silicon and glass gratings are able to align the liquid crystal along the grating direction for film thick-nesses above 15μm, in the grating period range 664nm - 24μm, as determined by using both x-ray diffraction and optical microscopy. The size of the top layer and the grating period range are of the order of magnitude of the correlation length for the smectic liquid crystal film. In this study, the region just above the critical thickness in smA droplets was studied as a function of temperature beyond the smA - N transition. Special attention was given to the region of thickness around 40μm, a region where the competition between the air-liquid crystal interface and the liquid crystal-substrate interface was most pronounced. We observed a persistence of molecular order and alignment in the layer along the gratings through the smA - N transition.

INTRODUCTION

The need to confine LC materials inside low dimensional geometries requires an understanding of how LC's interact with different substrate materials, as well as the different methods used to prepare these substrates. The ordering in LC films is in general stronger than in bulk samples; for example, in freely suspended films a quasi-smectic phase appears[1]. We have previously found[2-5] that both silicon and glass gratings are able to align the liquid crystal along the grating direction for film thicknesses above 15μm, in the grating period range 664nm - 24μm, as determined by using both x-ray diffraction and optical microscopy. This result set upper and lower boundaries for grating preparation. The results of our measurements were fit to a multi-layer orientational model in order to determine the thickness of the observed aligned layer. In addition, we found that for films thicker than the critical thickness, an approximately 10μm layer at the top of the film remained under the influence of the air-liquid crystal interface, which forces the molecules in the liquid crystal to align perpendicular to the surface of the film and the glass substrate.

In this contribution we are concerned with the behavior and evolution of order in smectic LC films in contact with solid grated substrates, such as those described above, as a function of temperature. This is an issue of both fundamental and applied importance. Specifically, the region near a LC phase transition is of great interest. The persistence of smectic-like ordered regions beyond a phase transition has been observed in standing LC films[1,6,7], as well as encased smC*[8-11] and smA films, and smC* droplets in contact with diversely prepared substrates[12-15]. This effect is associated with memory effects as well as over-all temperature stability in LC device applications[8]. Fundamentally, the behavior of LC crystals with solid substrates as a function of temperature illustrate theories of wetting and critical behavior[16]. The molecular orientation within this transition region depends on the chemical nature of the substrate and the substrate coating as well as the competition of molecular versus aligning forces inside the LC film[2-5,17]. The competition of aligning forces produces re-orientation transitions just below the the nematic-isotropic LC transition in certain weak anchoring substrates. These transitions are associated with the disordering of the molecular arrangement at the LC-solid substrate inter-face[18,19]. LC materials with a smA-isotropic LC transition re-orient in the presence of a strong aligning substrate when transition from the smA phase progresses from the bulk of the film[19]. The re-orientation of the LC molecules at the surface affects the overall orientation of the bulk film[15]. Preliminary optical measurements in our films indicated the persistence of large domains aligned along the direction of the grating grooves through the nematic-smA and the nematic-isotropic phase transitions[5].

We present a preliminary analysis of structural studies on the possible persistence of smA ordering in 8CB films deposited into grated glass substrates, close to the LC-substrate interface, in the vicinity of the smA-nematic transition.

EXPERIMENTAL

As in previous studies, octylcyanobiphenyl (8CB) was chosen because it exists in the smectic A phase at room temperature and it has high chemical stability. Gratings with a period of 10μm were produced on soda lime glass microscope slides using conventional photolithography method in a class 1000 clean room, from a mask generated on a Mann 3000 pattern generator. Details of the etching process appear in reference 2. The gratings have dimensions of 1cm x 1cm and a channel depth of approxiamtely 2-3μm. To achieve temperature control, a heating element approximately 1000Å thick was deposited on the opposite side of the gratings. This heater con-sisted of two concentric nichrome rings, with diameters of 7mm and 9mm, respectively. The rings were connected to a bonding pad con-

sisting of a 3000Å film of Aluminum. A DC voltage of about 25V drove the heater to approximately 80°C with a current of 30mA. A chromel-alumel microthermocouple was evaporated along the edges and center of the gratings. A negative of the thermocouple mask was made on a ferrous oxide plate. Positive resist was spun and soft baked on clean slides; the resist was exposed through the ferrous oxide mask. A 1000Å film of chromel was deposited onto the substrate, using an electron beam evaporator set at a pressure of 10^{-6} millibar. The undesired metal was removed by soaking the slides in acetone. This process was repeated to deposit a 1500Å film of alumel. The evaporation changes the ratio of metals comprising the two alloys, which requires that each thermocouple be individually calibrated. 8CB films were prepared in-situ, according to the methods described in references 2 and 5, using a slow temperature cycling into the isotropic.

The x-ray scattering experiments were performed at the National Synchrotron Light Source, beamline X22B, using 9.5keV (1.305Å) x-rays, focused on a spot of $2mm^2$ at the sample position. The detector slits were kept at a 10(vertical) x 4(horizontal)mm^2 opening. The diffraction experiment has been described previously[2]. The samples were mounted on a four circle Huber goniometer. In order to study the orientation of the smectic layers in the films, we performed both out-of-plane (001) as well as in-plane (100) Bragg scans. The former gives information on the smectic layers aligned parallel to the plane of the substrate, whereas the latter gives information on the smectic layers aligned perpendicular to the plane of the substrate (molecules parallel to the substrate). The grating direction was chosen to coincide with the (100) direction on the plane. We performed a series of χ scans on the films to observe the evolution of the in-plane peaks in the film. The diffraction angles θ,2θ were held constant at the Bragg value for 8CB. At $\chi = 90°$, the observed scattering corresponds to smectic layers parallel to the surface of the gratings (molecules perpendicular to the plane of the substrate). Conversely, at $\chi = 0°$, the signal observed corresponds to smectic layers aligned perpendicular to the plane of the substrate (molecules parallel to the plane of the substrate). The nominal angle of incidence α in these runs was 0° for 9.5keV x-rays. Finally, we performed azimuthal ϕ-scans at $\chi = 0°$ in order to observe the degree of in-plane disorder. For these scans, $\phi = 0°$ corresponds to the [100] direction.

RESULTS AND DISCUSSION

Figure 1 shows the results of Bragg scans along the grating direction (100) for a 44μm film deposited on a 10μm period grating prepared as described above, as a function of temperature, in the range 22°C - 35°C. Films of this thickness exhibit both a homeotropically aligned layer at the LC-air interface as well as a homogeneously aligned layer above the

LC-substrate interface[2,4,5]. We note that wheareas the intensity of the in-plane peak is six times smaller at 34°C than at 22°C, the full width at half maximum (FWHM) increases by only $1.125q_o$, where $q_o = 0.195Å^{-1}$ for 9.5keV x-rays. The FWHM is approximately $0.08q_o$ at 22°C, and $0.1q_o$ at 34°C, and is almost constant in the 22°C - 32°C range. The reciprocal of the FWHM is proportional to the coherence of the ordered domains within the layer along the grating direction. This coherence length does not change appreciably over temperature, persisting at temperatures greater than 0.5°C above the smA-nematic transition for 8CB. This suggests the presence of smA like regions beyond the transition temperature.

The intensity of the (100) peaks is associated with the size and crystallinity of the homogeneously aligned layer domains. A similar decrease in peak intensity is observed in the out-of-plane (001) peaks (not shown). To ascertain the evolution of the entire film as a function of temperature, we performed χ scans as described above.

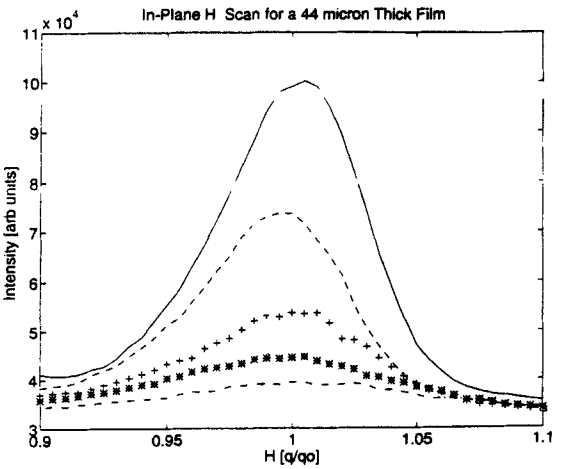

FIGURE 1. X-ray diffraction scans along the direction of the gratings (100) for a 44μm film as a function of temperature:
——: 22°C; - - - : 30°C; +++: 32°C; *** : 34°C; - · - ·: 35°C.

Figure 2 shows the evolution of the χ scans as a function of temperature. Note that there is an increase in the diffraction signal in the region between $\chi = 20°$ and $\chi = 65°$ as a function of temperature. This increase may indicate a development of ordered regions within the sample where the molecules are tilted with respect to the plane of the substrate. They may also indicate the appearance of a disordered region in the center of the sample. Such a region, whether tilted or disordered, increases with increasing temperature. Thus the intensity of the in-plane (100) peak is also associated with the thickness of the homogeneously aligned layer within the film. The results from the χ scans suggest that the film

disorders from the center. Both in-plane as well as out-of-plane peaks are observed even past the nematic-isotropic transition. We are currently performing a more detailed numerical analysis of our results in order to ascertain the nature of the central region in the film, as well as the thickness of the homogeneous layer as a function of temperature.

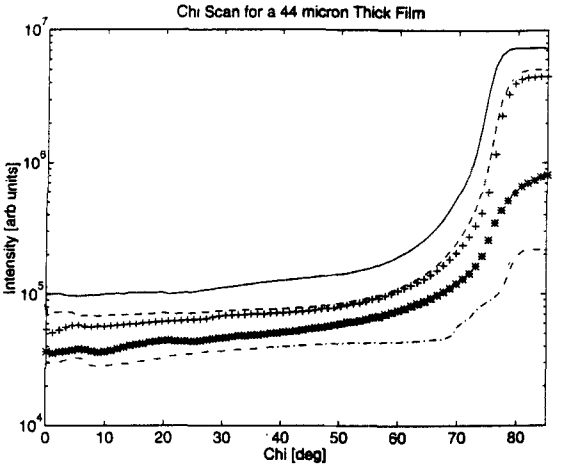

FIGURE 2. χ scans for a 44μm film as a function of temperature:
——: 22°C; - - -: 30°C; +++: 32°C; ***: 34°C; - · - ·: 35°C.

The results of the azimuthal ϕ scans appear in Figure 3. The samples were prepared using a slow cooling cycle which results in alignment of the LC molecules along the grating direction, $\phi = 0°$. The azimuthal spread, which is approximately 40°, persists as a function of temperature through the smA-nematic transition, as seen in Figure 3.

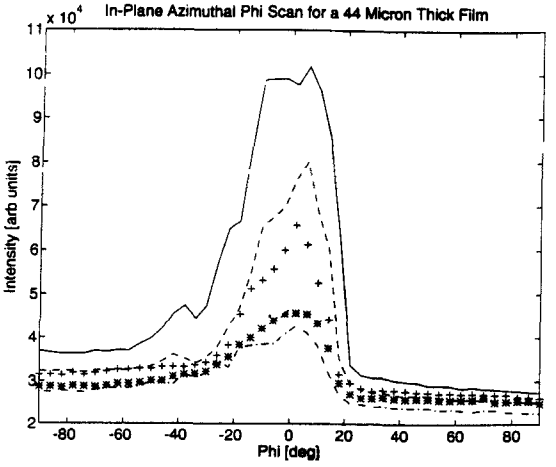

FIGURE 3. ϕ scans for a 44μm film as a function of temperature:
——: 22°C; - - -: 30°C; +++: 32°C; ***: 34°C; - · - ·: 35°C.

The grating repeat period exceeds the size usually required to align and orient nematic LC's[20]. These results, coupled to the presence of an in-plane peak, may indicate the presence of smA regions well above the smA-nematic transition, and even close to the nematic-isotropic transition. These regions are apparently located close to the LC-substrate.

CONCLUSIONS

We have presented a preliminary analysis of the effect of temperature variation on 8CB films deposited on 10μm repeat period glass gratings. We observed an apparent persistence of in-plane ordering above the smA-nematic transition, and close to the nematic-isotropic transition, as manifested by the presence of an in-plane diffraction peak. This may be associated with the presence of smA ordered regions close to the LC - glass substrate interface. The decrease of the in-plane diffraction peak signal is coupled to an increase in the χ scans of the diffraction signal associated with $\chi = 20° - 65°$. This result may indicate the development of either a tilted or a disordered region within the film. It also suggests a mechanism of film disordering which begins at the center of the film. Finally, the molecular alignment along the gratings persists well above the smA-nematic phase transition as seen in the ϕ scans.

Work in progress includes the analysis of our results to determine the evolution of the size of the homogeneous and homeotropic layers within the film, as well as to ascertain the nature of the intermediate layer, as a function of temperature. Future work will concentrate on the immed-iate region close to the smA-nematic transition.

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