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# Molecular Crystals and Liquid Crystals

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## LIQUID CRYSTAL ALIGNMENT ON REPLICATED NANOSTRUCTURED SURFACES

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Interaction of liquid crystals with nanostructured surfaces is studied experimentally. Whereas photolithographic or AFM methods were used in the past for surface structuring, we use besides photolithography essentially replication methods for the creation of well-controlled nano-topologies. We established a laboratory process to prepare surface structures that can be as large as  $4 \times 4$  inch and allow preparing test cells in a production like environment. We combined different methods and tested for soft embossing and direct writing in photoresist. Soft embossing was made with UV curable materials. The direct writing in photoresist was done by means of laser beam interferometry. Structure parameters were changed and the surfaces were characterized by surface anchoring energy measurements and by electro-optical switching behaviour. The obtained alignment is quite uniform over large surface areas despite of a certain variation of the surface parameters. In the case of interferometrically produced grating structures with periods ranging from 350–800 nm strong anchoring was observed and no pretilt could be found.

Keywords: displays; liquid crystal alignment; nanostructures; replication

#### INTRODUCTION

Liquid crystal alignment is usually induced with rubbed polymer films [1,2]. Nowadays, nanostructured gratings on large surfaces and preparation technologies allow new alignment concepts using nanostructured surfaces.

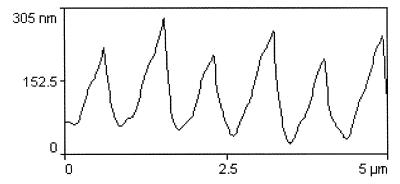
Our work was partially financed by the CTI. We thank C. Rockstuhl for providing the transmission data for nanostructured gratings.

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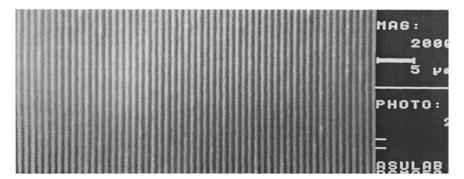
Such method might be used to align liquid crystal in displays [3–9]. New features have to be added that improve the display performance remarkably [10–14]. For this, it is important to find a practical way of fabricating such structures on large surfaces areas that is compatible with conventional liquid crystal technology. One important aspect is the surface quality over larger areas. Because of the elastic properties of the liquid crystal and the thickness that is much larger than the structuring of the surface it is expected that replication can be used for fabrication.

#### SURFACE MANUFACTURING AND CHARACTERIZATION

Fabrication of nanostructured surfaces can be done in different ways. Hot embossing and UV (often called cold or soft) embossing processes are used. Heat and pressure are used in hot embossing to replicate a surface structure in a thin plastic film. In our case we used UV embossing with photo-curable glue that can be polymerised at room temperature. A silicone (Polydimethylsiloxane PDMS) mould was fabricated by using master grating of 350 nm and 800 nm period with heights of 200 and 300 nm respectively. The master gratings were made by laser beam interference in photoresist. A nickel protection layer on the photoresist prevents from technological difficulties when the PDMS mould is formed at higher than room temperature. The active areas were the surface grating is present are  $20 \times 30$  mm. The mould is  $4 \times 4$  square inch and a few millimeters thick. UV curable glue from Norland was dissolved in acetone with different concentration ranging from 10 to 40% and spun on the surface substrates. Spinning speed was varied between 3000 and 5000 rpm, which results in thickness of the glue, layer between 0.2 and 2.5 µm. The replication



**FIGURE 1** AFM surface profile of a UV embossed replicated surface. The master was a sinusoidal grating. Deformations are visible indicating the difficulties in the reproduction process.



**FIGURE 2** SEM image of a replicated nanostructure shows the quality over larger areas. The image shows a surface area of  $43\mu\text{m} \times 20\mu\text{m}$  approximately.

is made by laminating the PDMS stamp on substrates with controlled pressure. A 350 Watt mercury discharge lamp needs about half a minute to completely polymerise the alignment layer. After UV curing the stamp was removed. A first quality check can be made by visual inspection. For a grating period of 800 nm and a height of 300 nm light diffraction allows judging on uniformity and defects. If there were already defects visible, the grating surface was not further processed. The quality of the replicated surface was additionally checked by atomic force microscopy (AFM) and scanning electron microscopy (SEM) as shown in Figures 1 and 2. In general the quality depends very much on the processing conditions like thickness of the UV glue layer and pressure. With the developed replication machine all parameters can be well controlled and the resulting nanostructured gratings were very well suited for liquid crystal alignment.

#### ALIGNMENT OF LIQUID CRYSTAL AND CHARACTERIZATION

Cells with nanostructured surfaces as alignment layers are fabricated since long time ago [4,9]. It is known that such surface substrates could give strong anchoring and good alignment condition. We made planar parallel and twist cells and inspected the alignment quality by naked eye and under the polarization microscope. Cells with conventionally rubbed polyimide on one or both substrate surfaces were taken as reference. Their properties where compared and the quality of the polyimide cells is set as standard. Three type of cells were used: standard cells with rubbed polyimide PI2545 (DuPont) (called polyimide-polyimide), cells with one grating substrate and one rubbed polyimide substrate surface (called

polyimide-grating), cells with gratings on both substrate surfaces (called grating-grating).

For all our replicated gratings, the nematic director aligns spontaneously with the grating direction i.e. perpendicular to the grating vector. This seems to be the energetically favored alignment [3] although the aspect ratio (ratio of height to period) for some grating structures approaches 2/3. For planar parallel grating-grating cells and for the polyimide-grating systems the liquid crystal is well aligned. Small variations of local nematic directors appear as domains of different extinction directions. The difference of orientation of the optical axis is estimated to be smaller than 2°. In polyimide-grating cells, the pretilt angle of polyimide surfaces and a chiral dopant help to implement stable mono-domain structures for twisted nematic cells. Such cells have a very high alignment quality. In the 90° twist cell of the grating-grating type, domains are visible that indicate different directions of the twist sense. This is an indication for very low pretilt angles. With a chiral dopant such problems can be avoided and the cells show good uniformity.

#### AZIMUTHAL ANCHORING ENERGY

Measurements of the azimuthal anchoring energy were performed by using a method that allows determination of the anchoring strength without knowledge of refractive index of the liquid crystal [15–17]. The method is based on analysis of the twist angle in a twisted nematic cell with varying thickness. Due to the twist deformation, a surface torque is exercised on both surface substrates. The smaller the cell thickness becomes the larger the torque. If the torque is strong enough, it changes the orientation of the director at the surface i.e. the azimuthal angle. That makes the twist angle changing and can be detected by looking for a minimum of the transmitted intensity between linear polarizers. In the experiment, the angle of minimum transmission is measured for different thickness while the thickness of the liquid crystal layer can be calculated from the cell geometry. The method depends very much on the texture quality. If there is a superstructure or a microscopically structuring of the surface substrate present the method fails. For strong anchoring conditions on different substrate surfaces a change of the twist angle cannot be detected and the measurement does not allow to determine azimuthal anchoring conditions. One has to be very careful by evaluating the range for which the method applies. Under the condition of strong anchoring on one substrate surface or of equal anchoring conditions of both surfaces the azimuthal anchoring energy can be calculated [18]. A plan-convex lens with a radius of curvature of  $R = 387.6 \,\mathrm{mm}$  was treated with an alignment polymer PI 2545 (Dupont)

**TABLE I** Measured Azimuthal Surface Anchoring Energy for Different Substrate Surfaces

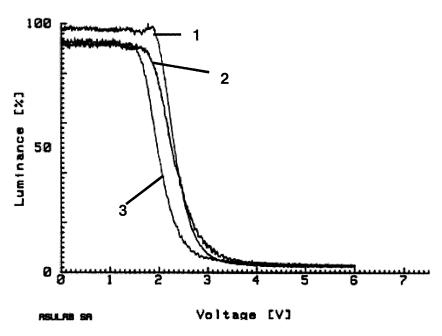
Surface material	Treatment	Azimuthal anchoring energy [J/m²]
Polyimide PI 2545	Rubbing	$5.4 \times 10^{-3}$
Norland NOA 68	$\begin{array}{c} \text{UV embossing} \\ p = 800\text{nm},d = 300\text{nm} \end{array}$	$6.5 \times 10^{-5}$

and rubbed to assure high anchoring strength. It was placed on the substrate under investigation to form a 90° twist cell. The liquid crystal mixture E7 (Merck) was used and orients spontaneously. For very thick liquid crystal layers, at the outer areas of the lens, the relaxation took a few minutes to become uniform and defect free. Results of the measurements are summarized in Table I. The anchoring energy for the polyimide is about two order of magnitude higher than for the nanostructured surfaces. In general the values are typical for high surface anchoring energies.

#### **ELECTRO-OPTICAL PERFORMANCE**

Planar and twisted nematic cells were fabricated and electro-optic characterization was made. The voltage dependence of the luminance of a twist cell between crossed polarizers is shown in Figure 3. The threshold voltage of the cells was found to be about  $2\,V_{\rm rms}$  for an  $8\,\mu m$  thick cell filled with E7 driven with 300 Hz AC voltage. The threshold for polyimide-grating cells is about the same as for polyimide-polyimide cells and no evident shift caused by the residual dielectric layer was found here. In our case the threshold for smaller grating period was smallest (curve 3), and even smaller than the reference value for polyimide-polyimide (curve 1). The situation is different when the grating-grating structures are investigated. As seen in Figure 4, a remarkable increase of about 0.5 V of the threshold appears which is most likely due to the residual dielectric barrier at the surface. Because of the lack of a pretilt angle, the switching is often not uniform and domains are created for medium voltages. For small pretilt a sharp onset of the switching behavior of the cell is expected. That was not found in the switching curve 4 shown in Figure 4. That leads to the conclusion that the alignment is microscopically not uniform or the surface polar anchoring of the liquid crystal is weak.

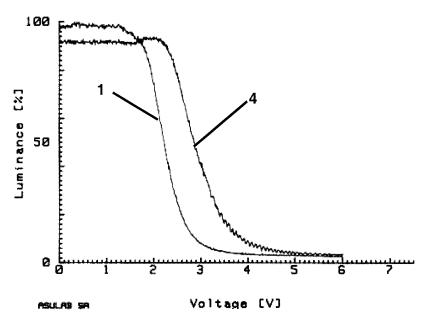
To have an idea about the time dependence of switching properties the switching-ON and OFF for a step function of  $5\,\mathrm{V}$  ( $\mathrm{V_{rms}}$  at  $300\,\mathrm{Hz}$ ) was recorded between crossed polarizers. Figure 5 show the corresponding curves. The switching-ON curve for the grating-grating system (curve 2)



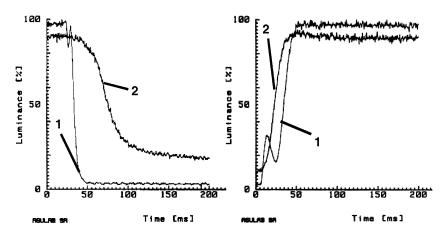
**FIGURE 3** Electro-optical response curves of twisted nematic cells. The luminance is measured as a function of the applied voltage. A reference cell (curve 1) with standard polyimide alignment is compared with cells were a polyimide-grating combination is used for alignment. Two different grating periods of 800 nm (curve 2) and 350 nm (curve 3) are shown.

indicates slow switching with switching time twice as long as for a polyimide-polyimide cell (curve 1) recorded at the same voltage step. Switching OFF the grating-grating cell is fast and reliable as can be seen in Figure 5 right. That indicates strong anchoring conditions.

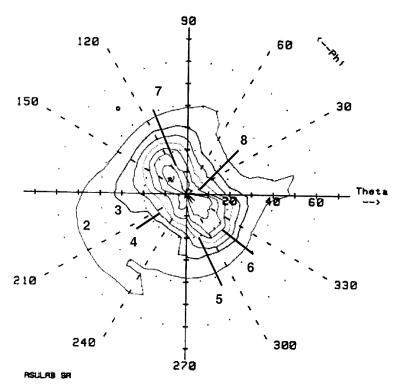
A draw back of the grating aligned cells is the transmission and contrast properties. The reference cell aligned with polyimide has a contrast ratio higher than 1:34 and the grating aligned cells give approximately 1:26. The angle dependence of contrast ratio and luminance are main parameters for high quality displays. Figures 6 and 7 shows those quantities for a grating period of 350 nm and a structure height of 200 nm. The cell thickness is 8 µm and the cell was filled with E7 (Merck). The curves were recorded with an automated Autronic-Melchers system. In Figure 6 the luminance contrast ratio is plotted as a function of the observation angle. The maximum contrast ratio is 9 and decreases rapidly with increasing polar angles. Looking a the shape of the iso-contrast curves in Figure 6 it becomes evident that the typical off axis maximum of the contrast doesn't exist anymore which indicates again low pretilt angles. Iso-luminance



**FIGURE 4** Luminance as a function of voltage for a grating-grating aligned cell (curve 4) compared to a standard polyimide cell (curve 1). In the case when two gratings are used the residual dielectric layers lead to a remarkable increase of the critical field.

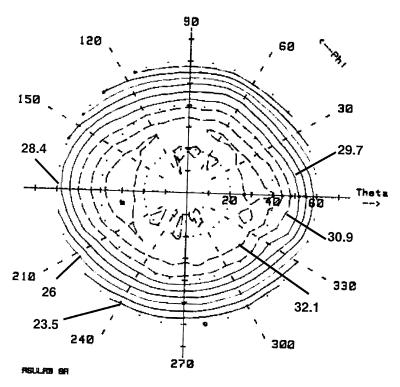


**FIGURE 5** The switching behaviour of grating aligned twisted nematic cells (curve 2) are shown in comparison to standard polyimide cell technology (curve 1). The twisted nematic cells were aligned with gratings on both substrates surfaces (grating-grating) with 350 nm period and 200 nm height. Left: Switching ON from 0 to 5 V. Right: Switching OFF.



**FIGURE 6** Measured iso-luminance curves for a twisted nematic cells realized with two grating surfaces (grating-grating). The contrast ratio has a maximum in the centre of about 9. In the outer areas the contrast is less than 2 (0–5 V).

curves in Figure 7 show balanced system with maximum values of 32%. The transmission values for grating aligned cells are always lower than for polyimide reference cells. A grating-grating aligned twisted nematic liquid crystal cell with polarizers along the direction of the grating and with a grating period of 800 nm has a transmission of about 92% of that of a polyimide-polyimide reference cell for white light. To understand this, diffraction effects of the incorporated gratings on the transmission are simulated. Because grating periods are comparable with the wavelength transmission for the zero diffraction order has to be calculated with rigorous methods. Applying a quasi-analytic solution [19] allows exact calculation of zero order transmission for the whole spectra. Fresnel reflection losses are not taken into account. The grating material (Norland adhesive) has a refractive index of  $n_{\rm NOA}=1.52$ . The refractive indices of the liquid crystal are assumed to be  $n_{\rm e}=1.75$  (extraordinary) and  $n_{\rm o}=1.5$  (ordinary). At 550 nm and for a grating of 800 nm period and 300 nm height



**FIGURE 7** Measured iso-luminance curves for a grating-grating aligned twisted nematic liquid crystal cell. The luminance is uniform and starts at values about 32 in the centre.

embedded in the liquid crystal the transmission reduces to 99.3% for TM polarization. For TM polarization the light is polarized along the grating vector and the index difference is given by  $n_{\rm o}-n_{\rm NOA}$ . When TE polarization is used the transmission reduces to 90.4%. The index difference is now  $n_{\rm e}-n_{\rm NOA}$  and much higher as before. These are remarkable values and can explain the reduced transmission. For the luminance, values of 99.5% (TM polarization) and 93.2% (TE polarization) follow. Smaller gratings with 350 nm periods and 200 nm height cause less pronounced loss. The luminance transmission is 100% for TM and 95% for TE.

#### SUMMARY

We have investigated the alignment properties of replicated surfaces. Large surfaces can be replicated with UV embossing techniques with a sufficient quality to align liquid crystals. Anchoring conditions are strong and the alignment is good. Gratings with too wide periods give remarkable diffraction losses and should be avoided. The switching dynamics is changed because of residual dielectric layers at the surface. Usually the threshold increases. Because of a very small pretilt angle, domains appear that worsen the optical appearance and reducing the contrast ratio and viewing angle dependence when the cells are switched.

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