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# Molecular Crystals and Liquid Crystals

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## Multi-Configurations in Nematic Liquid Crystal Films: A Microrubbing Approach

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We report a technique for the patterned alignment of liquid crystals on polyimide using microrubbing ( $\mu$ -rubbing). Homeotropic polyimide was  $\mu$ -rubbed with a metallic sphere 1 mm in diameter under a specified load of 150 grams. We observed that  $\mu$ -rubbing induces a high pretilt planar alignment when homeotropic polyimide is used. Liquid crystal cells were constructed from two  $\mu$ -rubbed polyimide substrates, whose rubbing directions are oriented orthogonal to each other. At the intersection points of two  $\mu$ -rubbed lines, a twisted nematic configuration was observed. In the other sections of the sample, either a splay or homeotropic configuration is realized. A pretilt of  $10^\circ$  was measured in the  $\mu$ -rubbed area. The electro-optical performances of the twisted nematic regions were also investigated.

**Keywords:** homeotropic; liquid crystal displays; microrubbing; planar; polyimide; pretilt

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#### INTRODUCTION

Patterned alignment of liquid crystals has wide spread application in many industries, including flat panel displays [1-5], telecommunications [6] and security [7,8]. In the flat panel display field, patterned alignment is usually sought after to create a multidomain configuration to improve and widen the viewing angle [9-13]. Several patterning techniques that have been exploited for this purpose are based on either organic or inorganic films. These techniques include selfassembled monolayers [14], photopolymers [15], reverse rubbing of polyimide [5], oblique evaporation of SiO<sub>2</sub> [4,5] and amorphous inorganic films such as diamond like carbon [16]. More sophisticated approaches include patterning of polymer surfaces with atomic force microscope (AFM) tip, which under certain circumstances can create tristable memory effects [17]. In telecommunication applications, patterning is often used to create diffraction gratings. Rosenblatt and coworkers showed that AFM rubbing [18,19] could create a polarization grating [20]. Recently, Honma and coworkers showed that μ-rubbing could be used to manufacture polarization independent gratings [21]. Security applications utilizing liquid crystals are becoming an exciting new area [22]. Moia and coworkers showed that photopolymer could be used to create personalized photographs of faces only visible when viewed between crossed or parallel polarizers [23]. The large and growing application for micropatterning of liquid crystals will capture the attention of liquid crystal research for years to come.

Here we report the patterned alignment of liquid crystals using the  $\mu\text{-rubbing}$  technique that results in a planar alignment with a high degree of pretilt. The  $\mu\text{-rubbing}$  was performed on homeotropic polyimide. A metal ball 1 mm in diameter was used for the  $\mu\text{-rubbing}$  process under a suitable load with a specified transverse speed. We demonstrate that under one set of electrodes, a multi-configurational sample is created consisting of twisted nematic, splay and homeotropic regions.

#### **Experimental**

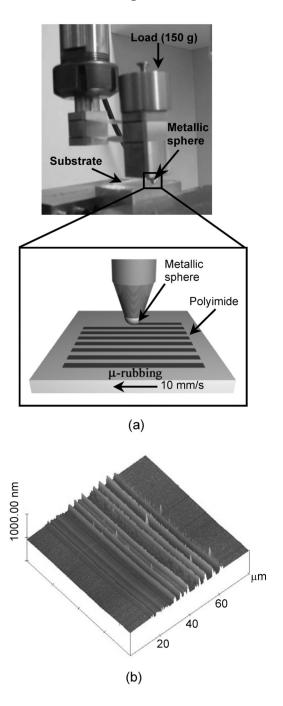
The polyimide precursor, A75114 was used to create a homeotropic orientation layer (JSR electronics) on indium tin oxide (ITO) coated glass substrates. Spin coating of the polyimide precursor was performed using a Karl Süss CT 62 spin coater (5 seconds at 1000 rpm, 40 seconds at 5000 rpm) on the ITO side of the  $25 \times 25$  mm substrates. After spin coating, the substrates were preheated to  $100^{\circ}$ C for 10 minutes. The samples were then imidized at  $180^{\circ}$ C for 90 minutes in a

vacuum oven. The thickness of the polyimide coating was  $\sim 100\,\mathrm{nm}.$  Liquid crystal E7 from Merck (Darmstadt, Germany) ( $T_{\mathrm{NI}}=58^{\circ}\mathrm{C},$   $\rho=1.06\,\mathrm{g/cm^3},~\epsilon_{//}=19$  and  $\epsilon_{\perp}=5.2,$  and  $\Delta n=0.225)$  was used for the cell configuration. The  $\mu\text{-rubbing}$  of polyimide substrates was carried out with a mechanical device using a 1 mm diameter metallic sphere. Patterns were recorded by writing at a constant load (150 g) and velocity (10 mm/s) at room temperature. The liquid crystal cell was constructed with the  $\mu\text{-rubbing}$  direction on the top and bottom surface oriented orthogonally and secured with UV curable glue (Norland UV Sealant 91). The cell gap was controlled by 5  $\mu\text{m}$  spacers (Merck). The cells are filled with liquid crystal material E7, by capillary action at 80°C, which is  $\sim 20^{\circ}\text{C}$  above the nematic-isotropic transition temperature of the LC.

The  $\mu$ -patterns in the polyimide were investigated by scanning probe microscopy (Nanoscope IIIa, Digital Instruments, Santa Barbara, California) equipped with conventional Si $_3$ N $_4$  cantilevers, measured in tapping mode. Optical micrographs of the liquid crystal configurations were recorded with polarized light microscopy (Zeiss LM Axioplan) equipped with a digital camera. The cell thickness was determined by UV-visible spectroscopy (Shimadzu UV-3102 PC). The electro-optical characteristics were investigated using DMS 703 display measuring system (Autronic-Melchers GmbH). A square AC signal of 1kHz was used to drive the cells for the dynamic response measurements. Pretilt measurements were carried out using Autronic, TBA 107 which employs the crystal rotation method. For this measurement antiparallel cell were constructed with 18  $\mu$ m cell gap and placed perpendicular to the rotating table of the instrument.

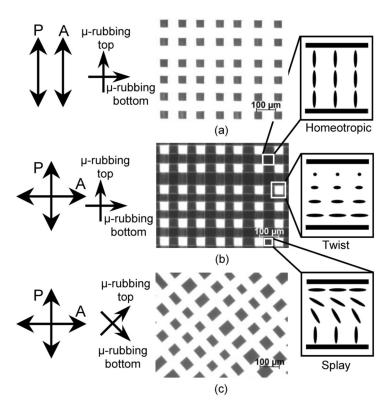
#### RESULTS AND DISCUSSION

Figure 1(a) shows the experimental apparatus used for the  $\mu$ -rubbing process. Several parallel lines were recorded at constant spacing between  $\mu$ -rubbed lines. The surface roughness of the metallic sphere was found to be 8 nm using AFM. During  $\mu$ -rubbing process microgrooves are formed on the polyimide, which may be one mechanism responsible for the observed unidirectional planar liquid crystal alignment. Figure 1(b) shows the atomic force microscopy image of the  $\mu$ -rubbed area (a load of 150 grams used). We obtain a line width of 47  $\mu$ m for a polyimide thickness of  $\sim$ 150 nm. The surface roughness of the polyimide is found to be 40 nm, which is five times larger than the surface roughness of the metallic ball. The depth of the microgrooves is on average between 20–30 nm.



**FIGURE 1** (a) A photograph of the  $\mu$ -rubbing apparatus used in this study and an illustration showing the  $\mu$ -rubbing tip apparatus. (b) An AFM image of the  $\mu$ -rubbed homeotropic polyimide at a load of 150 g.

In the data that follows, we show that the unrubbed polyimide results in homeotropic alignment as expected; however the  $\mu$ -rubbed region enforces planar alignment with a high surface pretilt angle. We propose the following physical mechanism for the modification of the alignment surface from homeotropic (before  $\mu$ -rubbing) to a planar alignment with large pretilt (after  $\mu$ -rubbing). Polyimides that are known to induce homeotropic anchoring are either doped with long chain aliphatics or are functionalized with long chain aliphatic molecules. When aliphatic chains pack on a substrate parallel to the substrate normal (highly aligned, compact assembly of aliphatic chains), they are known to induce homeotropic alignment of liquid crystal molecules coming in contact with them [24]. We conjecture that the m-rubbing process unidirectionally aligns the aliphatic chains at a

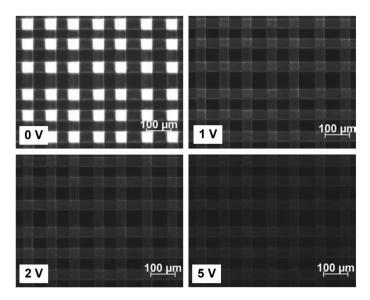


**FIGURE 2** Optical micrographs of liquid crystal cell from  $\mu$ -rubbed homeotropic polyimide with crossed patterns, (a) between parallel polarizers (b) crossed polarizers (c) cell at  $45^{\circ}$  between crossed polarizers. Three different liquid crystal configurations are also shown schematically.

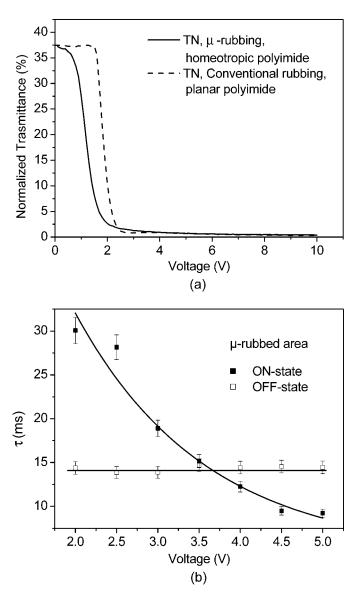
specific angle. This mechanism leads to a dramatic change in the anchoring direction for liquid crystal molecules interacting with the aliphatic-rich surface.

Liquid crystal cells were constructed with two  $\mu$ -rubbed substrates orthogonal to each other, where  $\mu$ -rubbing induces planar alignment of liquid crystals. Figure 2 shows the optical micrograph of the cell viewed between the polarizers. In the Figure 2 we can observe three different configurations. A twisted nematic configuration was observed where the two  $\mu$ -rubbed areas intersect on the top and bottom substrates. A splay configuration is observed in areas with hybrid anchoring (homeotropic polyimide on one substrate and  $\mu$ -rubbed polyimide on the other substrate). Finally a homeotropic configuration is observed in the unrubbed areas on both substrates as expected.

The switching behavior of the  $\mu$ -rubbed cell (twisted nematic region) is carried out by placing the cell between cross polarizers and applying a voltage from  $0V \to 5V$ . Figure 3 shows the optical micrographs of the cell under increasing voltage. The formation of disclination lines becomes more visible in the dark field (higher voltages). Figure 4(a) shows the electro-optical characteristics of the twisted nematic area formed by the  $\mu$ -rubbing process as compared to the twisted nematic



**FIGURE 3** Optical micrographs of a liquid crystal cell with homeotropic polyimide substrate having  $\mu$ -rubbed pattern on both sides which are orthogonal, shown between crossed polarizers at different voltages (0V  $\rightarrow$  5V).



**FIGURE 4** (a) Electro-optical characterization of the TN area of the  $\mu$ -rubbed patterns of homeotropic polyimide compared with conventional TN cell. (b) ON and OFF time of TN area of  $\mu$ -rubbed patterns, at a frequency of 1 kHz.

region created from the conventional velvet cloth rubbing. It can be seen from the graph that the switching voltage for the twisted nematic area resulting from  $\mu$ -rubbing is lower than that of a conventional

rubbed twisted nematic and an ON/OFF contrast ratio of 98:1 is observed. The near thresholdless nature of the  $\mu$ -rubbed region indicates that there is a high pretilt for the twisted nematic area formed from the homeotropic polyimide. This behavior is consistent with early models correlating pretilt to threshold voltages [25]. To verify this, we measured the pretilt of the  $\mu$ -rubbed region using the crystal rotation method and observed a 10° surface pretilt angle Figure 4(b) shows the dynamic response time of the twisted nematic area formed by the  $\mu$ -rubbing of homeotropic polyimide. The ON-state dynamic response time is defined as the elapsed time between 90% to 10% transmission. Conversely, the OFF-state time is the time elapsed between 10% to 90% transmission. The measurements were performed in the normally white TN mode. The OFF-state (relaxation) is essentially constant and the ON-state roughly scales as the inverse voltage as expected from theory [26].

#### **CONCLUSIONS**

We have presented recent results on patterning liquid crystals using a  $\mu$ -rubbing technique. We have shown how multi-configurational modes can be created consisting of twisted nematic, splay and homeotropic states. We have demonstrated that when polyimide that originally induces homeotropic alignment is  $\mu$ -rubbed, the  $\mu$ -rubbing induces a planar alignment with a high pretilt. The electro-optic performance results are consistent with this observation, showing a near thresholdless switching behavior. We believe this  $\mu$ -rubbing technique has value in many application areas, including displays, telecommunication and security. For practical display applications a multi-ball system (comblike) can be used to pattern large area surfaces. These are only preliminary results but we believe there is richness in physical phenomena in these systems making them exciting to further explore on a fundamental level. The subject of future study will correlate the applied load of μ-rubbing to the pretilt angle and surface molecular anchoring strength. In addition, we believe there is a strong correlation between applied load and the surface roughness as measured with AFM. These topics will be the subject of future publications.

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