

## Control of liquid crystal pretilt angle using polyimide mixture

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### Abstract

A mixture of two different types of polyimide, one having an alkyl side chain and ordinarily used for homeotropic alignment, and the other without a side chain and ordinarily used for planar alignment, is deposited and baked at high temperatures. When the resulting cell is filled with a nematic liquid crystal, it is found that the pretilt angle is a function of the baking temperature or mixing ratio, and can be controlled continuously over the range 0 to 90.

### 1. Introduction

During the past few years, much attention has been devoted to achieve control of a liquid crystal's pretilt angle. Several methods have been proposed. These include dipping a vertically aligning polyimide in a solvent [1], irradiating SiC layers of varying composition with an ion beam [2], and rubbing a polyimide that has a relatively rigid backbone designed for homeotropic alignment [3-7]. Recently, Yeung, *et al* [8] and Ho, *et al* [9] deposited a mixture of vertical and planar-aligning polyimide precursors onto a substrate. The resulting bulk tilt angle was controlled by means of relative concentrations of the two polymers.

Based on the concept of using a dual-polymer alignment layer to control the effective polar angle in the bulk, we extended our work on polyimides with side chains and examined mixtures of two Nissan Chemical Industries polyamic acids, the products SE-1211 and RN-1175. Unlike those used by Yeung *et al.*, we found that these materials do not undergo phase separation as the solvent evaporates, and that the resulting alignment layer does not form domains [10-11].

### 2. Experimental

#### Mixture preparation

We used a mixture of the commercially available polyamic acid SE-1211 and RN-1175, dissolved in varnish as supplied by the manufacturer, with the concentration of the polyamic acids being approximately 4% by weight of the total solution.

1) For baking temperature variation experiment, mixing ratio SE-1211:RN-1175 = 25:75 in weight was chosen. The mixture was spin coated onto ITO coated glass slides at 1800 rpm for 9 sec. The slides were prebaked at 80 °C for 30 min, and then were fully baked at temperatures ranging from below 230 °C to 290 °C for 50 min. These baking temperatures are significantly higher than that prescribed by the manufacturer (180 °C for 50 min). Overbaking the polyamic acids has two effects: It further imidizes the backbones, thereby promoting nearly planar alignment, and cleaves away a fraction of the side chains of the SE-1211 component, thereby weakening the tendency for vertical alignment.

2) For mixing ratio variation experiment, baking temperature was set to 255 °C, which is sufficiently higher than the prescribed baking temperature. The mixtures were spin coated at 2000 rpm for 9 sec onto ITO coated glass substrates. The substrates first were prebaked at 110 °C for 3 min, and then fully baked at 255 °C for 25 min.

#### Cell fabrication

Both substrates were rubbed extremely weakly with a cotton cloth using a rubbing machine. The purpose of the rubbing was to promote a spatially homogeneous azimuthal orientation for the tilted liquid crystal, and the softness of the rubbing ensured that the rubbing did not alter the naturally occurring polar pretilt angle. Each pair of slides was placed together, separated by glass spacers dispersed in a UV curable epoxy, in an antiparallel configuration and the thickness was measured by interferometry. Nominal

cell spacing was around 10  $\mu\text{m}$ . The cells then were filled with a nematic liquid crystal 5CB(Merck).

### Pretilt angle measurement

To determine the pretilt angle  $\theta_0$  in the nematic phase, light from a He-Ne laser (wavelength  $\lambda=633$  nm) passed consecutively through a polarizer, a Babinet-Soleil compensator, the cell, an analyzer, and into a detector. The polarizer and analyzer make  $+45^\circ$  and  $-45^\circ$ , respectively, with the rubbed direction of the cell. Initially the Babinet-Soleil compensator was adjusted to minimize the transmittance in the absence of the cell. The cell then was reinserted into the optical path and a potential difference of 50 V at frequency 1 kHz was applied across the cell thickness; this drives the liquid crystal director orientation in the bulk to be parallel to the applied field (i.e., perpendicular to the substrates), except very close to the surfaces. On reducing the voltage of the cell continuously to zero and simultaneously adjusting the compensator, we determined the optical retardation of the cell at zero voltage  $\alpha \left[ \equiv 2\pi\Delta n_{\text{eff}}(\theta_0)d/\lambda \right]$ , where  $d$  is cell spacing and  $\Delta n_{\text{eff}}$  is the effective birefringence  $\Delta n_{e,f}(\theta_0) \equiv n_e^f(\theta_0) - n_o \left[ \cos^2 \theta_0 / n_o + \sin^2 \theta_0 / n_e \right]^{-1/2}$ . Here  $n_e = 1.719$  and  $n_o = 1.528$  are the extraordinary and ordinary refractive indices, respectively, of 5CB at room temperature. Thus the pretilt angles were deduced from measurements of retardation  $\alpha$ .

### 3. Results and discussion

Figure 1 shows the pretilt angle as a function of baking temperature [10]. As shown in the figure, a minimum baking temperature for the mixture is required before a pretilt angle off the vertical direction appears. This is due to the necessity of breaking off a sufficient fraction of side chains and sufficiently imidizing the backbone for a tilt transition to occur. Above this temperature the pretilt angle varies smoothly with baking temperature, reaching a pretilt angle  $\sim 8^\circ$ , which would correspond to planar alignment.

Figure 2 shows pretilt angle vs. weight ratio  $f$  of RN-1175.  $f = [\text{RN-1175}] / \{[\text{RN-1175}] + [\text{SE-1211}]\}$ . For small  $f$ , the pretilt angle is near  $90^\circ$ , as expected; here the alignment layer is composed entirely or mostly of the vertically-orienting polyimide SE-1211 [11]. As revealed in Fig. 1, the baking temperature of  $255^\circ\text{C}$  used in this experiment was above a minimum temperature  $T_{\text{min}}$  below which the orientation remains

vertical. At higher concentrations of RN-1175 the pretilt angle  $\theta_0$  decreases relatively smoothly to  $\theta_0 \approx 30^\circ$  at  $f = 0.67$ . For  $f < 0.67$  we observed a uniform texture using polarized optical microscopy, but for  $0.67 < f < 1.00$  we observed a textured appearance due to macroscopic phase separation of the polyimides, as confirmed by atomic force microscope. In this concentration region the measured pretilt angle cannot be trusted, as it was comprised of multiple domains each having a characteristic pretilt. Only at  $f = 1$ , for which no phase segregation is possible, was a uniform texture observed with a pretilt angle  $\theta_0 = 8^\circ$ , i.e., nearly planar.

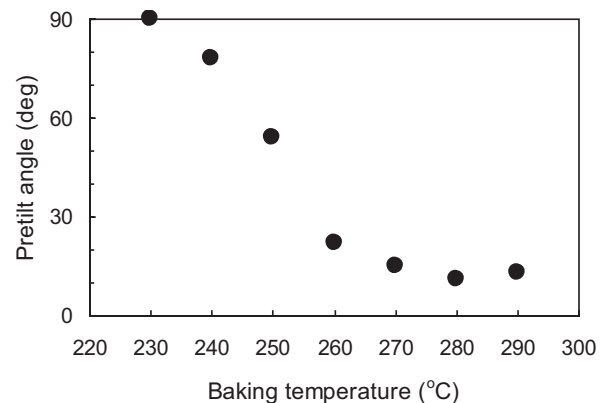


Fig. 1. Pretilt angle  $\theta_0$  vs. baking temperature.

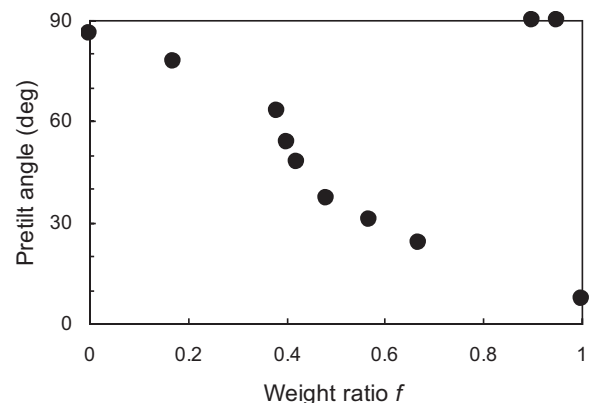


Fig. 2. Pretilt angle  $\theta_0$  vs.  $f$ , the weight fraction of RN-1175.

Unlike those used by Yeung *et al.* [8] who used atomic force microscopy to observe a very clear topography showing phase separation features on

length scales of several hundred nanometers with resolution of approximately 50 nm, we found that our mixtures do not undergo phase separation as the solvent evaporates, and that the resulting alignment layer does not form domains (except  $0.67 < f < 1.00$ ) [11]. Our observation is particularly interesting, given that the relative concentration of our two polyimides is not very dissimilar, in contrast to the phase separation observed by Yeung *et al.* at a much smaller concentration of vertically aligning polyimide in their experiment.

#### 4. Summary

In the paper we have demonstrated that the controlled pretilt of the liquid crystal can be achieved by a mixture of two different types of polyimide, one having an alkyl side chain and ordinarily used for homeotropic alignment and the other without a side chain and ordinarily used for planar alignment. By changing either the baking temperature or the mixing ratio, we obtained pretilt control over virtually full vertical alignment to planar alignment spectrum. The method can be easily extended to micropattern pretilt angles on a substrate.

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