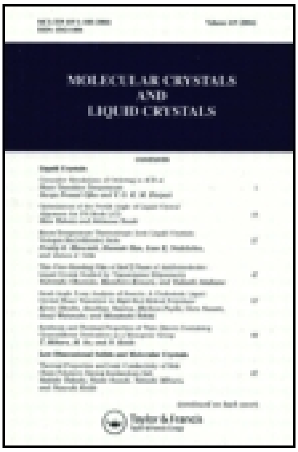

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Application of an Electro Spray Deposition (ESD) Method to Control the Pre-Tilt Angle in a Nematic LC Cell

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We applied the ESD technique to form an aligned LC layer with controlled pre-tilt angle. First, a horizontally aligned film was formed on the glass substrate by coating and rubbing. Then, a material promoting vertical alignment was sprayed using the ESD method. The aligned layer consisted of very fine domains mingled with horizontally and vertically aligned part. We were able to control the pre-tilt angle in a wide range by varying the amount of sprayed solution of the aligning material.

Keywords Electro-spray; LC alignment; pre-tilt angle

1. Introduction

A phenomenon when an electrostatically charged droplet is split into lots of microscopic particles by the Coulomb repulsive force has been known since long ago as an 'electro-spray'. At present this phenomenon is used as an ionizer in the mass spectroscope. One of the thin film formation techniques, called an electro-spray deposition (ESD) method, also applies this phenomenon. The technique was proposed by Morozov *et al.* in 1998 [1].

In the ESD method the microscopic particles are deposited on a charged surface of a substrate by the Coulomb force to form a thin film. The ESD method is important since particles with nanometer-scale order are obtained by an optimization of the spraying condition. Furthermore, the dried film can be formed directly in the atmospheric air.

The material prepared for spraying is first diluted in an appropriate solvent and next put into a thin capillary of the diameter less than 1 mm. A voltage of the order of kV is applied between the capillary and the electrically conductive substrate plate. Then, a cone-shaped liquid, called Taylor cone, arises at the top of capillary by the strong electric field concentrated at the capillary top, and is charged with same polarity as the capillary. The liquid spits out in the air as a droplet from the top of the Taylor cone by the Coulomb force. While moving the droplet continues to split into lots of microscopic droplets by the Coulomb repulsive force. After reaching the substrate these minute droplets are deposited on the substrate's surface by the

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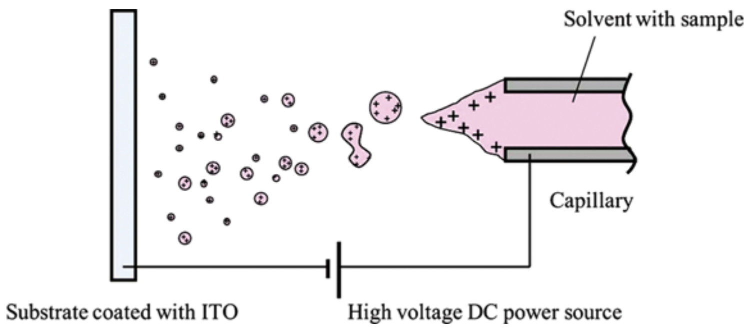

Figure 1. Principle of the ESD method. (Figure appears in color online.)

Coulomb attractive forces, forming a thin film. In some cases the solvent evaporates in a drying process, before the droplets reach the substrate.

In this study, the ESD technique is applied to form the alignment layer with controlled pre-tilt angle at the surface. The process goes in two steps. First, the horizontal alignment film is being formed on the glass substrate, coated with ITO and rubbed. After that, the vertically aligning material is sprayed with the ESD method. We are able to control the pre-tilt angle in a wide range by varying the amount of the sprayed solution.

So far, for controlling the pre-tilt angle, the spin-coated method of F. Sze-Yan Yeung *et al.* [2] has been used in which a mixture of horizontally and vertically aligning materials is employed. However, it seems that with this method it is difficult to obtain a pre-tilt angle with high reproducibility and the mixed aligning materials of high stability. We think that our ESD method is free of these shortcomings.

2. An Appropriate Choice of the Substrate Holder

When a cubic block consisting of aluminum was used as a substrate holder, it appeared difficult to form a uniform surface film. A reason for that was a large

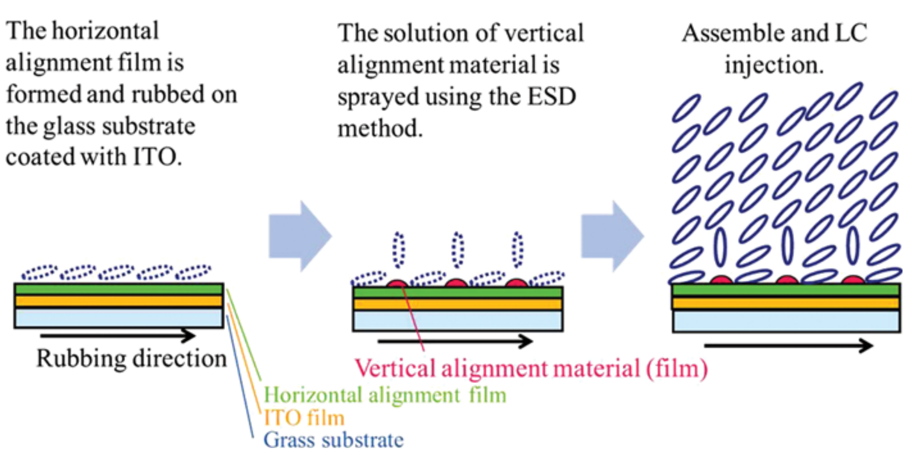

Figure 2. Scheme of the method to control the pre-tilt angle by using our ESD method. (Figure appears in color online.)

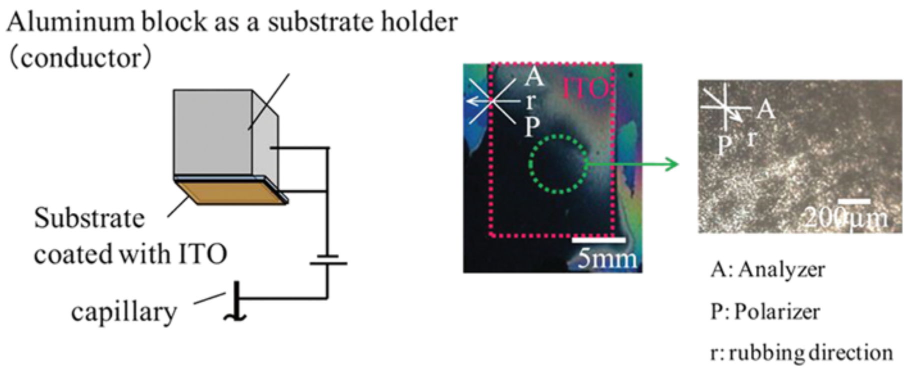

Figure 3. Conventional substrate holder and the photo of the LC cell. (Figure appears in color online.)

electric field at the corners of the aluminum block, as shown in Figure 3. So, the special substrate holder is necessary to utilize the ESD method for preparing a uniform film.

2.1. *Experiment*

Various substrate holders were used in the experiment, as shown in Figures 4(a) to (c). The best condition and configuration of the holder were determined by observing the LC orientational texture for each cell fabricated.

2.2. *Experimental Methods*

As already said before a first step in our studies was to prepare an ITO electrode on the glass substrate. The electrode has been patterned by the chemical etching, which

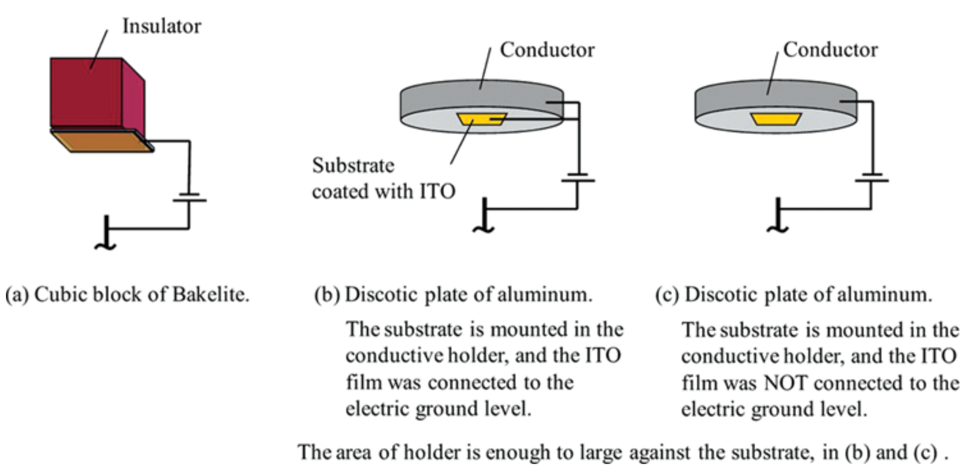

Figure 4. Three types of substrate holders used in experiment. (Figure appears in color online.)

enables to observe the LC ordering due to an electric charge concentration around the electrode's edge. Then the film of PI-A (Nissan Chemi. Ind.) of 4 wt% with horizontal alignment of the molecules was formed on the glass substrate, coated with a patterned ITO and rubbed. After that, the vertically aligning material SE-1211 (Nissan Chemi. Ind.) of 4 wt%, diluted by dichloromethane (DCM) in proportion SE-1211:DCM = 1:9, was sprayed by the ESD method, at the spray rate of 75 nl/s. The amount of total sprayed solution was 4 μl; the external diameter of capillary was 75 μm; the distance between the capillary top and the substrate was 6 cm, and the voltage of 7 kV with the electric field intensity of 1.2 kV/cm was applied.

Three types of substrate holders were employed in the experiment, as shown in Figures 4(a) to (c). In Figure 4(a), a cubic block made of Bakelite is used as a holder of the insulating material. Figure 4(b) shows the glass substrate mounted on the conductive aluminum discotic plate; the ITO electrode on the glass substrate is connected to the electric ground level. In Figure 4(c) shown is the same configuration as in Figure 4(b) except that the ITO electrode is not grounded.

For each holder the alignment material was deposited on the substrate surface by the ESD method. Then, the cell was combined with the other glass substrate on which the vertically aligned SE-1211 film was formed. The 5CB nematic LC was filled into the cell with thickness of 6 μm.

2.3. *Results and Discussion*

A direct view of each cell with crossed polarizers and under the polarizing optical microscope for the center and the edge of each cell are shown in Figure 5. Figures 5(a), (b), and (c) correspond to the cases in which the substrate holders used are shown in Figure 4(a), (b), and (c), respectively.

In the case when the substrate holder made of insulator was used, the vertically aligning material was deposited on the ITO electrode area using the scheme 4(a). Results are shown in Figure 5(a).

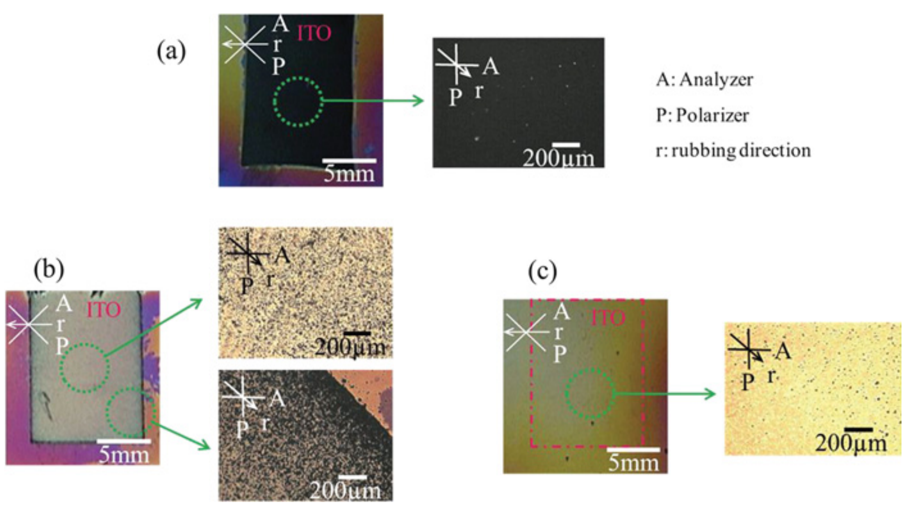

Figure 5. Photos of LC cells under crossed polarizers. (Figure appears in color online.)

In the case when the aluminum substrate holder was used and the ITO film was grounded, the same tendency was observed as in the case 5(a), see Figure 5(b). However, we noticed that the deposited material was concentrated around the edge of the patterned ITO film, as shown in Figure 5(b). Furthermore, the amount of the deposited material was reduced as compared to the case 5(a), because part of it was also deposited on the metal substrate holder.

Finally, when the aluminum substrate holder was used and the ITO film was not grounded, the black dots of the vertically aligned parts were not observed, see Figure 5(c). Only when the cell was studied with the polarizing microscope some black dots started to appear. We speculate that in this case the particles deposited on the aluminum plate.

From those results, we conclude that all types of holders shown in Figures 4(a) to 4(c) are adequate to deposit molecules uniformly with our ESD method. It seems that the best type of the holder is that shown in Figure 4(c), which is made of the conductive discotic plate with the glass substrate mounted on it and with the non-grounded ITO electrode.

3. Control of Pre-Tilt Angle

3.1. *Experiment*

We tried to control the pre-tilt angle by depositing the vertically aligning material with ESD technique on the substrate coated with horizontally aligned film. The substrate holder used this experiment was the one shown in Figure 4(c).

3.2. *Experimental Methods*

The same setup was implemented as one described in Section 2.2 (see Fig. 6). The spray rate was kept at 175 nl/s and the capillary of external diameter of 75 μm

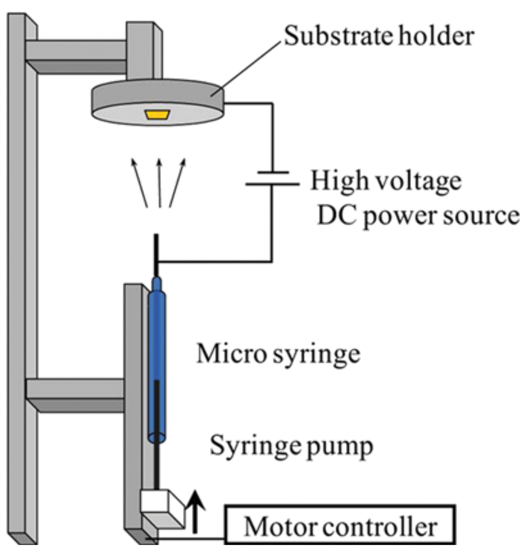

Figure 6. Scheme of our ESD implementation. (Figure appears in color online.)

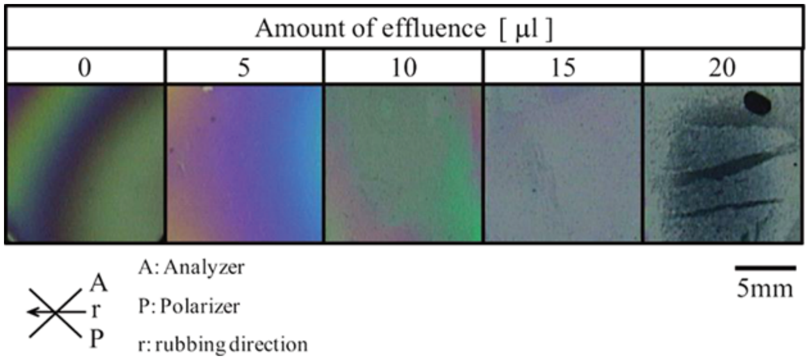

Figure 7. Photos of LC cells under crossed polarizers. (Figure appears in color online.)

was used. The amount of sprayed solution was 0, 5, 10, 15, and 20 μl. The cell was fabricated in the anti-parallel configuration with two substrates conducted by same aligning treatment; the cell of 6 μm and 20 μm thickness was used and the nematic LC (5CB) was injected into that cell. For the 20 μm thick cell the pre-tilt angle was measured by the magnetic null method [3]; the orientational texture for the 6 μm thick cell was scrutinized under crossed polarizers.

3.3. *Results and Discussion*

The snapshots of the cells are shown in Figure 7. Note that effluents of 20 μm cause that uniformity of the texture is lost.

Figure 8 shows the photos of the middle part of each cell of Figure 7, taken under the microscope with crossed polarizers. The vertically aligned region is observed as a black spot. The spot's size increases as the effluents of the vertically aligning solution is increased. For the effluents of 15 and 20 μl, the black spots of several tens of microns are observed. They disappear for the effluences of 5 and 10 μl.

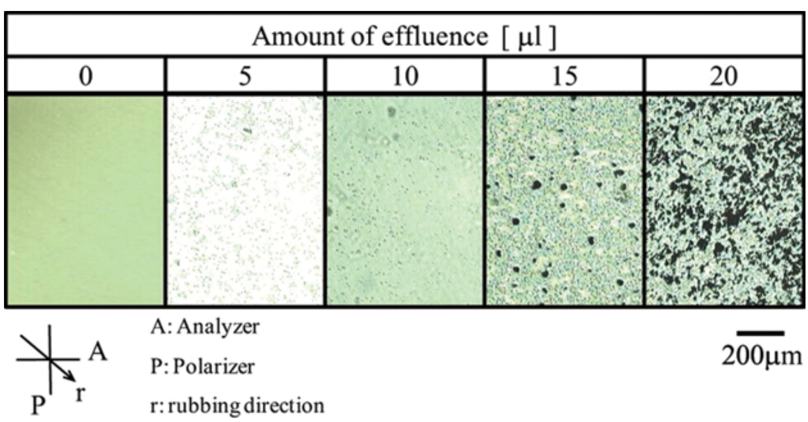

Figure 8. Photos of LC cells under the polarized optical microscope. (Figure appears in color online.)

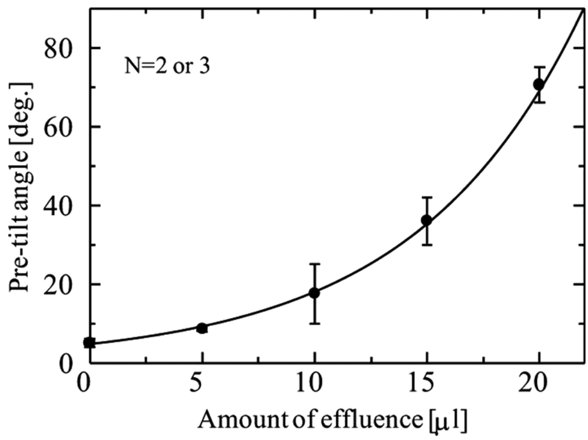

Figure 9. Measurement results for the pre-tilt angles.

The measurements for the pre-tilt angle are shown in Figure 9. The angle from 5 to 70 degrees was obtained, with the error not exceeding 5 deg. The reason why the reproducibility of the data deteriorates lies in a direct contact of the sample with air and the interfusion of the air bubbles in the syringe, or solution. In the case when the effluence of 20 μl is reached, the inhomogeneity of the texture can cause the error for the pre-tilt angle estimates to increase further.

4. Conclusions and Perspectives

We proposed the ESD technique to form the aligned LC layer with controlled pre-tilt angle. It is clear that the deposition of aligned LC material was affected by the shape and material of the substrate holder. The best condition for having the uniformly aligned film was obtained when the substrate holder was made of aluminum and the ITO film on the substrate was not grounded.

With the ESD technique we were able to control the pre-tilt angle in a range from 5 to 70 deg., by varying the amount of effluence in spraying the solution.

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