Second wind of the oblique deposition method of liquid-crystal alignment: Ion-beam sputtering technique

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Abstract — Liquid-crystal (LC) alignment on SiO_x films produced by ion-beam sputtering deposition was comprehensively studied. The conditions for planar, tilted planar, homeotropic, and tilted homeotropic LC alignment of high uniformity were determined. The alignment photostability and aging issue are discussed. An original sputtering system based on the anode-layer source excelling in high reliability and quality of sputtered coatings were used. Because this system can be easily scaled up, the alignment treatment of the large-area alignment substrates, including those used in modern LCD manufacturing, can be realized. The advantages of the sputtering LC alignment technique, in comparison with its vapor-deposition predecessor, are described.

Keywords — *Liquid-crystal alignment, sputtering deposition, anode-layer source.*

1 Introduction

For the new generations of LCDs, the demands on liquid-crystal (LC) alignment are becoming more stringent. The alignment treatment should be satisfactory for rather sensitive surface electronic elements, fixed spacers, *etc.* Working with large-area substrates (1500×1600 mm^2 for the present 6th generation, and 1870×2200 mm^2 for the planned 7th generation) creates difficulties with the alignment uniformity for large substrate sizes. The strong development of projection displays, caused by recent achievements in LCOS technology, requires high alignment uniformity on the microscopic level. These types of devices also sets strong demands on the photostability of the alignment layers that can be drastically increased by substitution of the organic layers with inorganic layers. The traditional alignment procedure dealing with the rubbing of organic films in not able to satisfy the demands described above. This has caused a strong need for new alignment methods that overcome the intrinsic problems of rubbing while, simultaneously maintaining all advantages of this procedure.

Presently, the so-called particle beam alignment method shows the most promise. The "particle-beam alignment" is a generalized term of methods operating with beams of different particles (ions, neutrals, electrons and mixtures thereof). These particles enter the substrate obliquely to generate surface anisotropy, which is an obligatory condition of planar/tilted LC alignment. The particle-beam alignment methods can be divided in two groups.

In the first group, particle beams cause modification of the alignment films (etching, molecular bond breaking, ion implantation, *etc.*). The interest in ion-beam alignment, known for over several decades,[1,2] was excited by recent publications of the IMB group.[3,4] The following works described LC alignment for different alignment materials and gas carriers.[5–7] The process is extended to the beams of accelerated plasma and adapted for the alignment treatment of large-area substrates.[8,9] However, the ion/plasma-beam processing has limitations. It simply provides planar and low-tilt LC alignment, while tilted vertical alignment is realized only for the limited class of organic materials with insufficient reproducibility.[9] Moreover, the unsolved problem of alignment aging[8,10] seriously hampers technological utilization of this method.

For the other group of methods, particles are deposited on the substrate-forming alignment layer. This principle was suggested by Janning in 1972.[11] In his experiments, highly uniform LC alignment was observed on SiO_x films obtained by oblique vapor deposition (VD). Shortly after Janning, Urbach *et al.*[12] revealed that LC alignment on SiO_x films is rather sensitive to the angle of deposition α. In case of α, determined by the film's normal, greater than some critical angle α_c, the easy axis of the LC lies in the incidence plane of particle flux and tilts towards the direction of particle beam incidence with a pretilt angle θ greater than 10°. The value of α_c depends on the LC and ranges 70–80°. For the opposite case, $\alpha < \alpha_c$, planar alignment is realized with the easy axis perpendicular to the particles' incidence plane. By analogy with the particle-beam etching procedure,[8–10] we define these alignment types as the 1st and the 2nd alignment mode. Studying the problem in depth, Moncade *et al.*[13] noticed that transition from the 1st and 2nd mode in VD process is rather broad; instead of having a critical angle they revealed a range of angles. In this range, the two-fold degenerate LC alignment was observed with two axes of easy alignment symmetrical with respect to the incidence plane of particle beam. The described alignment features are common for liquid crystals with positive dielectric anisotropy. In contrast, for LC with $\Delta\varepsilon < 0$ only, the 1st alignment mode was detected.[14–16] Interestingly, depending on the

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angle of deposition, the easy axis deflection from the film's normal was observed in two different directions.[16] The value of deviation angle increased with the deposition angle and reached 40° at grazing deposition.

The alignment in both modes was of high quality with excellent uniformity on the macroscopic and microscopic scale. The tilted homeotropic alignment of LC with $\Delta\varepsilon < 0$ on SiO_x layers is very suitable for LCDs operating in VA mode, especially for LCOS devices. As a result of many efforts,[17–20] the pretilt angle of LC with $\Delta\varepsilon > 0$ on SiO_x substrates was reduced to the values needed for operation in conventional TN- and STN-LCD modes (θ = 3–5°). This shows high application potential of the oblique deposition technique. Nevertheless, it remains to be the technique for laboratory use. There are at least several reasons why its technological implantation has been restricted: (1) vapor-deposition technique cannot be easily adapted for large-area substrates and mass-production line; (2) it consumes a lot of energy; (3) the parameters of LC alignment are not sufficiently reproducible, especially for LC with $\Delta\varepsilon > 0$.

The new promise of this technology involves the ion-beam sputtering deposition (SD) process. In this process, the material is extracted from only the top layer of the material plate (target). It substantially lowers power consumption, as compared with vapor deposition. By using extended sputtering sources, one can produce coatings on the large-area films.[21] Finally, because of the substantial difference in the adatom's energy, sputtering deposition is not a simple alternative to vapor deposition, but a method of obtaining new functions of the alignment films. Despite a great deal of potential, the alignment properties of SD films are not discussed in detail.[22–24] The majority of the results of this method are allowed only for internal use of the companies.

Our results of LC alignment on the films produced by the oblique sputtering deposition will be summarized. LC alignment modes, alignment uniformity issues, the dependence of the alignment parameters on the process conditions, and the alignment stability will be discussed. Finally, we draw conclusions about the most suitable application of this alignment technology.

2 Experimental

2.1 Sputtering deposition setup

The scheme of the sputtering source and deposition geometries are presented in Fig. 1. We used the sputtering device developed by Izovac, Ltd. It is based on the anode-layer ion source with the racetrack shape of the discharge area. It generates two sheet-like fluxes of Ar^+ ions* focused on the surface of a lengthy target with the dimensions of 60 × 500 mm^2. The incidence angle of ions onto the target, determined from the target's normal, is about 60°, which allows us to obtain the maximal efficiency of sputtering at the same power consumption. The ion-source aperture limits the flux of sputtered material providing its partial collimation.

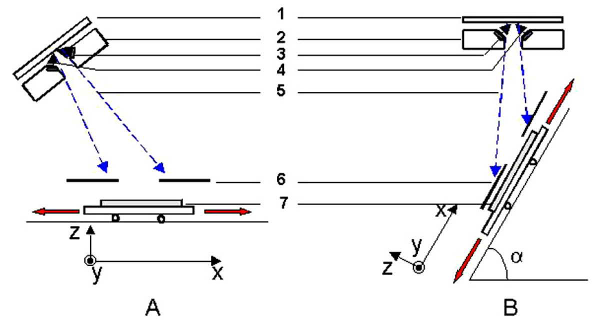

FIGURE 1 — Scheme of sputtering source and sputtering-deposition geometries. 1–3 — parts of sputtering source (1 – target; 2 – cathodes and magnet system; 3 – anode); 4 – ion beam; 5 – flow of sputtered particles; 6 – diaphragm; 7 – moving platform with substrates.

The SiO_x films were deposited on the substrates by sputtering the vitreous silica target by the argon-ion beam. To neutralize the charges generated on the non-conducting surface of the target under the action of the ion beam, the thermoionic compensator made of wolfram filament located near the target was used. In the majority of experiments, the parameters for the sputtering of the target material were maintained to be constant. In this standard regime, the residual pressure in the chamber was not higher than 3×10^{-3} Pa, the discharge voltage was 4 kV, and the discharge current was 400–450 mA. The thickness of the SiO_x coatings d_{SiO_x} was changed by the deposition time and measured by the quartz-crystal controller. We used 20 × 30-mm^2 glass slabs from Asahi Co., containing ITO electrode layers, as the substrates. Before SiO_x deposition, the ITO surface was cleaned with an anode-layer ion source. A section of the vacuum chamber with sputtering and etching devices is presented in Fig. 2.

To realize an oblique SD, two types of deposition geometries were used. For the first one, the sputtering system was rotated, while the glass slabs were set horizontally.

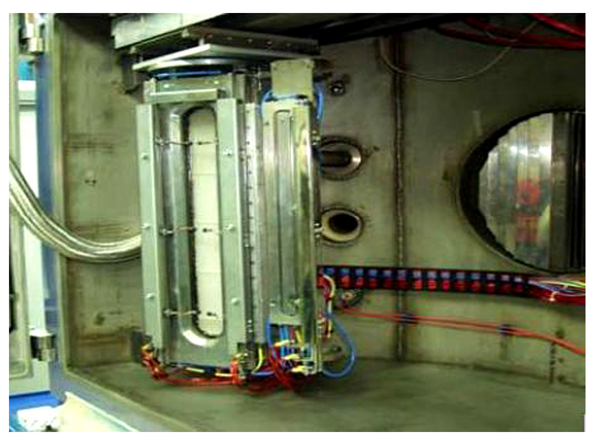

FIGURE 2 — Fragment of sputtering set up showing sputtering and etching sources.

*Precisely speaking, the anode layer source generates flux of accelerated plasma, and so it can be considered as a plasma source, Refs. 8–10.

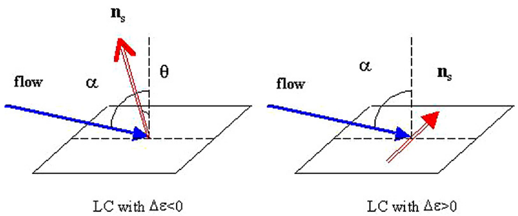

FIGURE 3 — Scheme showing orientation of LC easy axis $\mathbf{n}_s$ on SD SiO_x substrates. One can see that the LC with $\Delta\varepsilon < 0$ aligns in the 1st mode, while the LC with $\Delta\varepsilon > 0$ aligns in the 2nd mode.

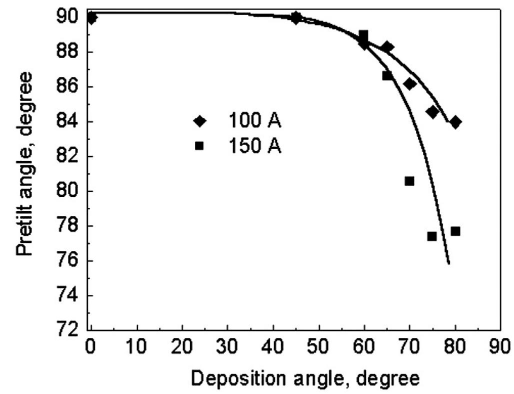

FIGURE 4 — Pretilt-angle *vs.* deposition angle curves for the SiO_x films of different thicknesses. Standard values of discharge voltage and discharge current. LC MLC6609.

The slot with a width of 2 cm served to limit the deviation of the deposition angle. The substrates were fastened onto the mobile platform and moved cyclically with a speed of 3 mm/sec. In the other arrangement, the sputtering system was mounted horizontally, while the substrates were set on an incline and cyclically scanned. These arrangements provided variation of deposition angle in the ranges of 0–75° and 0–85°, respectively.

2.2 Cells

For the alignment tests, two types of LC cells have been prepared: (1) the tested substrate contains a deposited SiO_x layer, while the reference substrate has a rubbed polyimide layer (asymmetric cells); and (2) both substrates contain SiO_x layers and assembled in an antiparallel fashion (symmetric cells). The asymmetric cells were used to determine the easy-axis direction on the SiO_x substrate (LC alignment mode). The symmetric cells were used to measure the pretilt angle by using the crystal rotation method. The cell gap was maintained by using spacers of 6 and 20 μm in diameter. We used LC with $\Delta\varepsilon > 0$ (E7, ZLI2293, ZLI4801-000, MLC12100-000 and TL216 from Merck) and LC with $\Delta\varepsilon < 0$ (MLC6608, MLC6609 from Merck and MJ961180 from Merck Japan). The in-plane uniformity of LC alignment was tested by sample observation using a viewing box or a polarizing microscope. The out-of-plane uniformity was judged from the measurement of the LC pretilt angle.

3 Results and discussion

3.1 Alignment modes

First of all, it should be noted that the properties of LC alignment are very weakly dependent on the type of deposition geometry. The key parameter is the deposition angle. Because of this, it will not make a difference between the results obtained for two types of deposition arrangements presented in Fig. 1.

The films produced by the oblique ion-beam deposition displayed uniform and stable alignment when the coating thickness exceeded 5–7 nm. However, the type of alignment depends on the sign of dielectric anisotropy of the liquid crystal. Figure 3 schematically shows the easy-axis direction of LC on SiO_x substrate for the case of LC with (a) negative and (b) positive dielectric anisotropy. For LC with $\Delta\varepsilon < 0$, the easy axis lies in the plane of particles incidence (1st alignment mode). The LC tilts towards the deposition direction, same as in case of grazing-angle vapor deposition.[16] This rule is for the entire range of deposition angles. For LC with $\Delta\varepsilon > 0$, the easy axis is perpendicular to the deposition plane (2nd alignment mode). The quality of LC alignment was excellent on a microscopic level for both types of LC.

Surprisingly, for LC with $\Delta\varepsilon > 0$, the 1st alignment mode was not observed for grazing-angle deposition (α = 80–85°), as in the case for the vapor-deposition technique. This difference might be explained by a different morphology of coatings, which, in turn, might be caused by a different particle energy for thermal-VD and ion-beam-SD techniques. Indeed, for the oblique deposition process, in which the film morphology is formed by the "self-shadowing effect,"[25] the structure should strongly depend on how well the adatoms move around on the substrate, *i.e.*, on the adatoms energy.[26] For thermal deposition, the adatoms have an energy of several milli electron volts, while sputtering deposition involves particles with energies of several electron volts and higher. The comparative studies of the morphology of VD and SD SiO_x films were carried out in Ref. 22, but, unfortunately, the range of deposition angles was not sufficiently wide to meet the conditions of the 1st mode for LC with $\Delta\varepsilon > 0$.

3.2 The alignment configurations for applications

The tilted homeotropic alignment realized for LC with $\Delta\varepsilon < 0$ makes sputtering deposition alignment technology rather attractive for LCDs operating in the VA mode. With this technique, the pretilt angle θ can be varied by several years. Figure 4 shows that, similar to the case of VD,[16] the pretilt angle gradually decreases with the angle of deposition starting from θ = 90° (homeotropic alignment) at α = 0°. The θ *vs.* α curve is sensitive to the thickness of the SiO_x layer d_{SiO_x}. As one can see from Fig. 5, the pretilt angle monotonically decreases with the thickness of SiO_x film with a

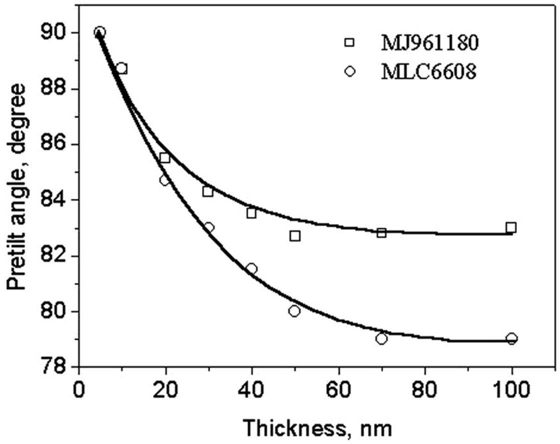

FIGURE 5 — Pretilt angle as a function of thickness of SiO_x coatings. Standard values of discharge voltage and discharge current. The angle of deposition is 85°.

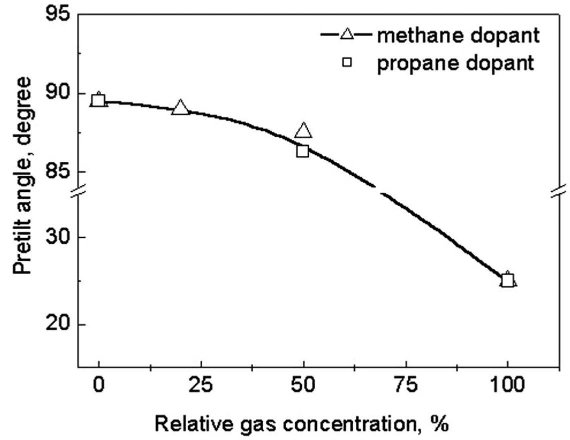

FIGURE 6 — Pretilt angle as a function of concentration of hydrocarbon gas when the mixture Ar/C_nH_{2n+2} (n = 1, 3) is used as a gas carrier in the sputtering system. The discharge voltage and discharge current are 2.5 kV and 100 mA, respectively. The thickness of the alignment films is 15 nm. LC MJ961180.

tendency of saturation. It is also sensitive to LC material. Note that the minimal value of the pretilt angle reached in our experiments is considerably higher than that which is obtained by using the VD technique (77° *vs.* 50°[16]). This might be also caused by the different morphologies of the films obtained by using the VD and SD methods.

The other possibility in controling pretilt angle is variation of the gas composition in the ion-beam sputtering process. Figure 6 shows that for the case of argon/hydrocarbon gas composition, the pretilt angle decreases with the concentration of hydrocarbon gas. For a pure methane or butane feed, a pretilt angle lower than 30° was achieved. The replacement of Ar on CF_4 also leads to a decrease in the pretilt angle and, simultaneously, to an increase in the rate of deposition. Apparently, the change in pretilt angle is caused by the incorporation of new types of atoms in the silicon oxide films and corresponding change of LC-substrate interaction.

To obtain the non-zero pretilt angle for LC with $\Delta\varepsilon > 0$, it is necessary to implement additional process to change the out-of-plane symmetry of the 2nd mode. This was realized by the two-step processing in two different ways. For the first case, we used the approach previously described for the VD alignment process.[17,18] The film was deposited by the flow incident in the y direction under the angle α_2, followed after the deposition in the x direction under the angle α_1 (Fig. 1). For a thickness of the second layer d_2 larger than 3–4 nm, the easy axis of the LC is perpendicular to the particles' incidence plane of the second deposition step (x direction). The latter means that the second coating overcomes the alignment action of the first coating. If $d_2 < 3$ nm, the easy axis lies in the yz plane and the pretilt angle is non-zero. The LC tilts towards the deposition direction in the second process. The problem is that the value of the pretilt angle does not exceed 2°. This is below the values required for the standard operation modes. More optimization work is needed to increase the pretilt angle and its reproducibility. The intriguing idea is to use different sputtering material on the second deposition stage, providing higher values for the LC pretilt angle.

In the second method, the deposition process is combined with the etching process. At first, the film is deposited in the x direction under the angle α_1 and then it is etched with the ion beam in y direction under α_2. The etching conditions were optimized to obtain good alignment properties. By using a low irradiation dose, one can break the alignment symmetry and induce tilted LC alignment with the easy axis in yz plane. The tilt direction corresponds to the direction of the ion beam. The pretilt angle is a non-monotonous function of etching dose. Figure 7 demonstrates this dependence for the O_2 working gas, but the same trend is observed for Ar. Evidently, the pretilt angle reaches 5° and then gradually decreases. After a certain dose level, which depends on the type of ions and their energy, the reorientation of LC in the

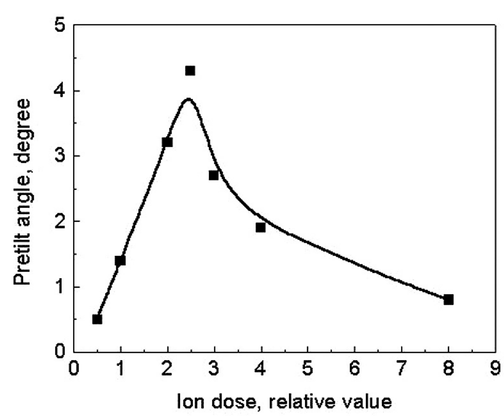

FIGURE 7 — Pretilt-angle values for the two-step processed films ("deposition plus etching" processing). The conditions of sputtering deposition: Ar as a gas carrier, $\alpha = 75°$, the standard values of discharge voltage and discharge current. The thickness of the deposited coating is 15 nm. The conditions of ion-beam etching: O_2 as a gas carrier, $\alpha = 75°$, exposure time is 3 min, discharge voltage is 1.7 kV, discharge current is varied in a range 15–100 mA. LC TL216.

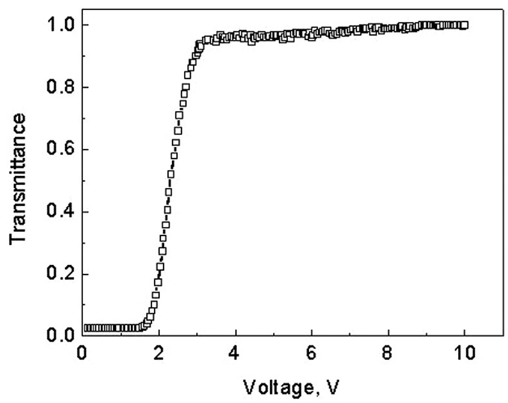

FIGURE 8 — Transmittance *vs.* voltage plot for VA cell (d = 6.4 µm) based on SiO_x alignment films. LC MJ961180.

perpendicular azimuthal direction is observed, in agreement with earlier observations for the etching alignment.[9,10] The reproducibility of the alignment characteristics in the second method is much higher than in the first method.

The other critical issue is the alignment uniformity. The pretilt-angle uniformity on the 20 × 30-mm^2 testing substrates is rather high; the pretilt angle deviation in different points of these substrates is not higher than 0.3° (LC with $\Delta\varepsilon > 0$) and 0.2° (LC with $\Delta\varepsilon < 0$), that is, practically, within accuracy of the measuring technique. By comparing the alignment on different testing substrates from the same processing set, we judged the alignment uniformity on the sample holder size (200 × 200 mm^2). The azimuthal and polar deviation of the easy axis was less than 2° and 0.5°, respectively.

The cells with both LC ($\Delta\varepsilon < 0$) and LC ($\Delta\varepsilon > 0$) demonstrate good electro-optic performance. As an example, Fig. 8 demonstrates a T–V curve of the VA cell. No defects appear in the cells under the LC reorientation in an electric field.

3.3 Alignment stability

Taking into account the alignment aging of the ion/plasma-beam alignment,[8,10] it is important to carry out aging tests for the SD films. According to the previous section, the most technologically attractive processes to date are the following: (1) one-step deposition for the tilted VA of LC with $\Delta\varepsilon < 0$; (2) two-step processing for low-tilt alignment of LC with $\Delta\varepsilon > 0$. These types of samples were chosen for aging monitoring. The alignment-quality and pretilt-angle aging in these cells at room conditions were investigated. No change in the quality of LC alignment (easy axis drift, appearance of disclination lines, *etc.*) was observed over the testing period. The typical aging curves of the pretilt angle are presented in Fig. 9. Apparently, that the pretilt angle of LC with $\Delta\varepsilon < 0$ is very stable. The pretilt angle of LC with $\Delta\varepsilon > 0$ on the layers obtained by the two-stage sputtering procedure is rather stable too. At the same time, it gradually decreases on the substrates obtained by the sputtering plus etching process.

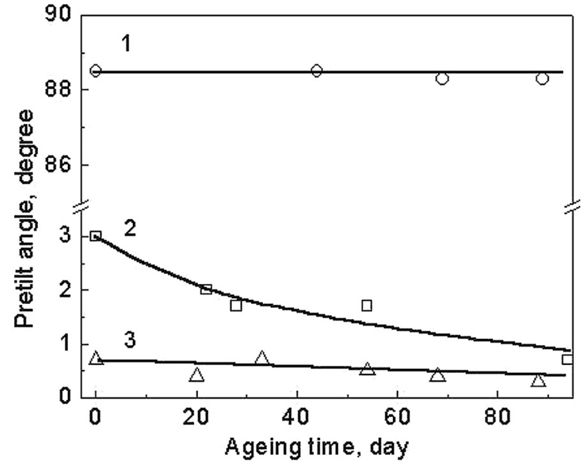

FIGURE 9 — Pretilt angle as a function of cell storage time. Room conditions storage. 1) One step deposition, LC MJ961180; 2) "deposition plus etching" processing, LC ZLI2293; 3) "deposition plus deposition" processing, LC ZLI2293.

A similar trend was earlier observed for simple etching alignment.[8,10] In the latter case, the alignment stability was substantially improved by passivation of the alignment films after alignment processing.[27,28] We believe that the same method can improve the alignment stability of the LC on the SD films subjected to anisotropic etching by the ion/plasma beam.

The samples with low-tilt alignment (θ = 2–4°, LC with $\Delta\varepsilon > 0$) on SiO_x substrates were also subjected to accelerated alignment photodegradation using the Philips testing line described in Ref. 29. We found that lifetime increases by a factor of 2.5 compared with the lifetime of samples based on standard rubbed polyimide layers. We believe that further improvement can be achieved by optimization of the LC, film coating conditions, and, possibly, surface modification with surfactants.[23]

4 Conclusions

According to results above, the sputtering deposition technique has great promise for modern LCD technology. It provides highly uniform alignment with controllable orientation of the LC easy axis in the azimuth and the polar plane. It is especially useful to realize tilted homeotropic (LC with $\Delta\varepsilon < 0$) and planar (LC with $\Delta\varepsilon > 0$) alignment of high photostability. The version of this technique used in the present work can be easily scaled up for the LC alignment on the large-area substrates, including substrates of modern generations. The advantages of the SD technique are demonstrated on examples of SiO_x films, but it allows for a large variety of aligning coatings with a wide spectrum of mechanical, electrical, and optical properties. Besides alignment films, the SD technique can be applied to produce other in-cell or out-of-cell functional layers (protective layers, antireflective layers, retardation films,[30] *etc.*) on the LCD plates. Our arrangement allows us to adapt this processes for large-area substrates and substrates of various shapes.[21]

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