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Glass Cleaning

Glass cleaning is one of the most important steps in liquid crystal display fabrication. Substrates are cleaned before processing and after many subsequent steps, and improper cleaning can result in

- electrical shorts
- cell gap variations
- poor alignment of the liquid crystal

to name a few fatal defects. It is therefore imperative to have an efficient cleaning process to remove all contamination from the glass surface.

- Contamination can be classified into two groups:
  - particulate
  - organic thin film

Particulate contamination can come from the operator (in the form of hair, skin flakes, bacteria, or clothing fibers), process equipment and supplies (flecks of dried photoresist, wiper fibers, dust, etc.), or the glass itself (fragments from cutting). Because typical LCD cell gaps are below ten microns, even a single particle a few microns in diameter can cause a fatal defect. For this reason, particulate contamination is the primary concern in the cleaning process.

Thin film contamination can be caused by improper cleaning and stripping procedures, or operator contamination, such as skin oils. These typically leave a thin organic film over all or a portion of the substrate, resulting in poor adhesion (ברקשת) or dewetting (淅溼), of subsequent coatings, and in some cases incomplete etching. After many procedures, a simple water rinse is not sufficient to remove process chemicals. Photoresist in particular can sometimes require ultrasonic agitation (מעונה) in an organic solvent for complete removal.

Cleaning methods

For LCD fabrication, a typical cleaning process involves ultrasonic cleaning with a mild (שומן) detergent (_metror) followed by a UV / ozone cleaning to remove organic contamination. Production environments will often use brush scrubbing or jet spray, with UV / ozone cleaning an option.

In ultrasonic cleaning, the bath is agitated at ultrasonic frequencies in order to dislodge (חריר) particulate contamination via cavitation (חרוור). The bath consists of deionized water with a neutral detergent, and is often heated to 50-60°C to aid in cleaning. Ultrasonic
cleaning is most effective on particles larger than 3-5 microns. After rinsing, the substrates are often dried using isopropyl alcohol.

UV/ozone cleaners are extremely effective on thin organic films. Short wavelength UV (below 300 nm) breaks down more complex organics, and the ozone reacts with the films to form carbon dioxide. UV/ozone cleaning is used as the final cleaning step. This method can be time consuming (5-20 minutes / substrate), and the substrates should move within minutes to the next process step. **UV/ozone cleaning improves adhesion of photoresists and polyimides.**

**Cleaning test**

The effectiveness of a cleaning process can be judged using several methods. The simplest of these is visual inspection, usually under UV light illumination. Ultraviolet light is scattered by particulate contamination, and thin films are readily visible, making it straightforward to judge the cleanliness of the substrate.

An excellent method for testing substrate cleanliness is by measuring contact angles with water. If a drop of water is placed on an inorganic surface, it should spread out and completely wet the surface. If there is an organic film on the surface, the water will tend to bead and form a contact angle close to 45°. This can be observed qualitatively by spraying the substrate with deionized water if properly cleaned, the water should sheet off of the glass rather than beading.

**Spincoating**

A popular method for the application of thin films is spincoating. The substrate is held flat by a chuck, the solution is poured onto the substrate, and the substrate is then spun at high speed for 20-30 seconds to obtain a uniform film. It seems counterintuitive that a high speed spin would give uniform film thickness, but the method works remarkably well. The main drawback to spincoating is the amount of material required for a single substrate--5-10 ml can be used on a single substrate.

**The concept behind spincoating**

As the substrate spins, the solution spreads to cover the entire substrate, and is pushed outward by the spinning. Ideally, the atmosphere above the substrate will saturate with solvent, preventing the film from drying before spinning is complete. When the spinning stops, the film relaxes back into a uniform thickness before being dried on a hot plate. Most spincoaters enclose the chuck (holder) in a bowl, which provides both safety and a barrier for the material which is spun off. The bowl, however, is usually stationary this means that turbulent air flow can kick up particles which can streak the substrate, and that the substrate will have a buildup of material in the corners due to the effect of wind. The ideal
solution is to have the bowl spin with the chuck this eliminates all effects of wind turbulence, and results in a uniform coating over the entire substrate.

There are several manufacturers (e.g. Solitec and Headway in U.S) of spincoating equipment. They provide units for small and large substrates with various degrees of automation. Some manufacturers supply spincoaters with the spinning bowl, but these units are considerably more expensive.

Some production environments use spincoating for photolithography, polyimide, and barrier layers, but printing is more desirable because of its more efficient materials usage. The disadvantage of printing is the high capital cost of the equipment (typically $500,000).

Roller coating

A popular method in small production environments is roller coating. This method can be used to coat photoresist for low resolution photolithography. The film uniformity is considerably less than for spinning or printing, but is adequate for direct addressed and other low resolution panels.

Dip coating

Other methods of coating include dip coating and meniscus coating. In dip coating, substrates are drawn out of a solution at a uniform speed. Both methods enjoy limited usage, but neither is even close to the spinning and printing methods.

Photolithography

The evolution in microelectronics technology, and microlithography in particular, has progressed at an astonishing rate. The conventional photolithography, which uses 365-405 nm irradiation, were able to print 0.5-0.6 mm features in production in the 1990s. Advances in optics have enabled exposure by shorter and shorter wavelengths. Indeed, photolithography using 248 and 193 nm light promises to dominate production technology well into the next century.

In semiconductor industry, the three-dimensional circuit elements are fabricated by a series of process collectively known as "lithography". The pattern is first generated in a polymeric film on a device "wafer", and this pattern is then transferred via etching into the underlying thin film. Diazonaphthoquinone-novlac materials will most likely remain the materials of choice for production of these devices. The costs of introducing new resist materials and new hardware are strong driving forces pushing photolithography to its absolute limit.

The technological alternatives to conventional photolithography are largely the same as they were a decade ago, that is,

- near- and deep-UV photolithography
- scanning electron beam lithography
- X-ray lithography.

The leading candidate for the production of devices with features as small as 0.3 mm is deep-UV lithography. The polymer that are used as radiation-sensitive resist films must be
carefully designed to meet the specific requirements of the lithography technology and device process. Although these requirements vary according to the radiation sources and device process, properties such as sensitivity, contrast, resolution, etching resistance, shelf life, and purity are ubiquitous.

In display applications high resolution has been made possible by decreasing the minimum feature size of the circuit element. The conventional means of increasing the resolution, that is, of increasing the circuit density, has been to make the active elements in the devices smaller, thereby increasing the number of active circuits that can be accommodated on a given area of display.

Electrode patterns

In order to pattern the electrodes on each substrate, a photolithographic process is used. This is the same process that is used in printed circuit board and integrated circuit fabrication. Each substrate is coated with a photosensitive material (photoresist), and selective areas are exposed to UV light (this pattern is generated by a photomask). A developing process leaves resist only on the desired electrodes. The substrate is then placed in an etch bath, which etches the ITO (Indium Tin Oxide) from the areas not covered by resist. After stripping of the photoresist and cleaning, the substrate is ready for subsequent processing.

Photoresists can be either positive or negative working. Positive resists, which include most commonly used liquid resists, are initially slightly soluble in a developer solution. Upon exposure to UV light, they become highly soluble. Therefore a 45-60 second developing with agitation creates a positive image of the photomask. Negative resists are often dry; that is, they are laminated onto the substrate rather than spun. These dry resists do not have high resolution capabilities, but are highly suited to a production environment. Developing is done in a chamber where the developer solution is sprayed on the substrate.

Resist Materials

<table>
<thead>
<tr>
<th>Type</th>
<th>Characteristics</th>
<th>Mechanism</th>
<th>Developer</th>
<th>Ad./Disadv.</th>
<th>Sensitivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Negative resist</td>
<td>less soluble in developer</td>
<td>crosslinking</td>
<td>organic solvent</td>
<td>image distortion</td>
<td>high</td>
</tr>
<tr>
<td>Positive resist</td>
<td>more soluble in developer</td>
<td>chain scission (حساس, הפרדה, חיתוך) or polarity change</td>
<td>aqueous solvent</td>
<td>high resolution</td>
<td>dry etch resistance</td>
</tr>
</tbody>
</table>

FIGURE 2: ITO electrode pattern.
Negative resists

Negative resists are a class of materials that become less soluble in a developer after exposure to radiation. Generally, the chemistry of negative resists involves some form of radiation induced crosslinking. The parent polymers are usually soluble in organic solvents, which in turn are used as developers. Materials include vinyl, epoxy, halogen containing polymers.

FIGURE 3

Positive resists

Materials that exhibit enhanced solubility after exposure to radiation are defined as positive resists. The mechanism of positive resist action in most of these materials involves either main chain scission or a polarity change. Ordinarily, the chain scission mechanism is only operable at photon wavelengths below 300 nm where the energy is sufficient to break main chain bonds. The best known of these so called dissolution (בריסת, פירוד, פירוק) resists is "conventional positive photoresist; a photosensitive material uses a novolac (פנול-פורמלdehyd) resin with a diazonaphthoquinone photoactive compound as a dissolution inhibitor. Upon irradiation, the diazonaphthoquinone undergoes a Wolff rearrangement followed by hydrolysis to generate a base soluble indene carboxylic acid.

FIGURE 4

As device features move into the submicron regime, advanced processing techniques and new lithographic technologies will need to accommodate high resolution, high-aspect ratio imaging over device topography. This necessitates the development of new resist materials with improved etching resistance, resolution and sensitivity. A recent developed sensitivity improving process involves the concept of chemical amplification. The chemical amplification principle has been used to design a number of negative resists based on acid catalyzed cation (חיובי) polymerization of appropriate monomers (a relatively light, simple organic molecule that can join in long chains with other molecules to form a more complex molecule or polymer) or crosslinking of polymers.

Photomasks

Photomasks can be of several types depending on the resolution & durability required, and the type of exposure system. Most mask aligners require chrome on glass masks—these are more expensive ($500-1500 typical), but provide good durability and high resolution. Mylar emulsion masks provide medium to low resolution (down to about 25 microns), but are cheap (less than $50) and can be used in many contact printers.
Radiation sources

Mercury arc lamps are the most popular light source for photolithography. Light in the 350-450 nm range is most effective with conventional photoresists used for medium resolution patterning. When resolutions below several microns are required, quasi-monochromatic light (g- and i-line) is often used, or shorter wavelengths are used. Since it is difficult to reliably etch ITO features smaller than 5-10 microns, conventional mercury lamps suffice for most LCD applications (Thin Film Transistor - TFT’s typically require the greatest resolution).

Exposure systems

A variety of exposure systems are available. Most production lines employ some type of step and repeat photolithography equipment for maximum throughput. In research and prototyping settings, mask aligners are most common. These allow the mask to be aligned with the substrate and/or previously placed features on the substrate before exposure. This is most important when doing multiple masking steps, such as those necessary for color filter and TFT fabrication. Many mask aligners allow both proximity and vacuum contact printing. Proximity printing increases mask lifetime, and decreases need for mask cleaning; because mask aligners have collimated sources, the substrate can be held a short (10-15 microns) distance from the mask without sacrificing much in accuracy. Contact printing is necessary for accurate gaps below 15 microns or so.

Low resolution exposure systems consist of a rubber mat, on which the substrate and Mylar mask are placed, covered by flat glass. The air between the mat and glass is evacuated, resulting in vacuum contact between the mask and substrate. UV exposure can then be carried out. These systems offer ease of use and low cost, but accurate alignment is not possible, and mylar masks must be used.

Liquid Resist Method

Photoresist is spun on the substrate at 3500-4000 rpm for 30 seconds. This results in a film thickness of about 2 microns. After spincoating, the substrate is placed on the 95°C hotplate for 90 seconds. Substrates are exposed on NuArc exposure unit (low resolution) for 20.0 units.

Process

Developers for most positive liquid resists are aqueous alkaline solutions (tetramethyl-ammonium hydroxide, TMAH, in this case). The substrate is immersed in the developer solution and gently agitated for 45-60 seconds. The substrate is then rinsed and blown dry with nitrogen.

Before etching, a hard bake at 115°C for 2-4 minutes is required. It is important that all liquid has been blown off of the substrate before hard bake; if not, it is very common to have a film residue which may prevent etching.
Donnelly recommends etching their ITO coatings in an acid bath of HCl:H2O:HNO3 heated to 55°C. Substrates are immersed in this bath for 90 seconds then rinsed.

Substrates are then soaked in KOH solution (to neutralize surface) for 60 seconds. A full cleaning is then necessary before proceeding to polyimide application.

**Surface Alignment**

In the liquid crystal devices, one of the most important problems is the surface alignment of the liquid crystal molecules. There are four basic surface alignments as shown below.

In practical application, a small tilt from parallel and perpendicular as shown in figures (c) and (d), namely, pretilt is important for obtaining domain-free orientation under electric field.


- The dispersion force is considered as the only alignment factor.
- It is assumed that the LCs align perpendicular to the free surface.

Mechanisms of the parallel, perpendicular and tilted homogenous alignments

**Parallel Alignment**

Parallel alignment is usually obtained as long as the surface is microscopically flat and liquid crystal does not contain amphiphilic impurity as well as surface polarity is too low to absorb the impurity. Normally, surface coated with fluorinated material (toxic reactive chemical element: a toxic pale yellow gaseous element of the halogen group that is the most reactive and oxidizing agent known. Source: fluorite, cryolite. Use: water treatment, making fluorides and fluorocarbons) gives low surface energy. Therefore, stable parallel alignment is obtained by decreasing the surface polarity by coating polymer or surface coupling agent, of which molecules tend to adsorb parallel to the surface. However, these alignment are random parallel alignment. In order to obtain homogenous alignment, unidirectional rubbing is necessary.
Mechanism of the alignment parallel to the rubbing direction is analyzed by Berreman [Phys. Rev. Lett., 28, 1683 (1972)].

**Perpendicular Alignment**

There are three proposed alignment mechanisms to obtain perpendicular alignment. Amphiphilic materials (surfactants) assisted alignment, i.e., amphiphilic material absorbs perpendicular to the polar surface and LC aligns according to the amphiphilic material. The second mechanism is the use of surface coupling agents such as silanes (silicon-hydrogen compound: a compound of silicon and hydrogen belonging to a group analogous to the paraffin hydrocarbons. Formula: $\text{Si}_n\text{H}_{2n+2}$) with long alkyl chains. The third mechanism is microscopic columnar structure-assisted alignment which is obtained by SiO-rotatively oblique (אלכסוני) evaporation as reported by Hiroshima et al. [Japan. J. Appl. Phys., 21, L791 (1982)]. The three alignment mechanisms are illustrated below. Materials and process for liquid crystals alignment in LCDs Alignment on the clean inorganic surfaces It has been known empirically that some liquid crystals align perpendicular to inorganic smooth surface such as $\text{In}_2\text{O}_3$ film. The reproducibility and uniformity of this type of alignment is poor as the substrate surface is ill defined. The cleaning procedures employed in the substrate preparation also play a role, e.g., MBBA (limited to Schiff bases) molecules will align perpendicular to the surface of acid treated glasses of oxides, but non-uniform alignment parallel to the substrate surface is obtained with fired or detergent cleaned glass. Oxidation of $\text{In}_2\text{O}_3$ coating in an oxygen plasma lead to layers causing parallel alignment of biphenyls. [G. Sprokel and R. M. Gibson, J. Electrochem. Soc., 124, 557 (1977)]

In general, the polarity of various metal oxides increases the tendency of perpendicular alignment of LCs increase. Weak surface polarity of metal oxides favors the parallel alignment. A table summarized by Uchida is given below.

Alignment on the surfaces organic polymers

**Polymer coatings on glass substrates can be employed to align liquid crystals**, but film uniformity and the substrate used influence the observed results. Several methods have been used to form the polymer layer which is preferably thin in order to avoid an excessive potential drop in the dielectric layer. The film may be transferred to the surface from a liquid, whereas polymer casting and thermal or plasma polymerization of the monomer have also been used. The most common method is to form the polymer from partially polymerized solution by dipping or spin coating followed by curing. Polyimide layers are the most popular polymers in LCD manufacturing to align LCs parallel with some pretilt. Polymer coatings do not sustain high temperatures are not suitable as the alignment layers.

In general, polymers orient the nematic director parallel to the substrate but do not induce uniform, reproducible LC alignment. Polyimides became popular in the early 1980's as LCD alignment layers, and their ease of use, stability, and reproducible results have made them the industry standard. Most polyimides are not easily dissolved (gamma-butyrolactone is a common solvent) and do not spincoat well. Because of this, they
are usually supplied in a precursor (chemical compound preceding another: a chemical compound that leads to another, usually more stable, product in a series of connected reactions) solution of polyamic acid and an organic solvent such as NMP. These solutions are highly hygroscopic (absorbing moisture: capable of easily absorbing moisture, e.g. from the air), able to absorb several percent of their weight in water within several hours; for this reason, they are typically stored in tightly sealed containers in a freezer. After spincoating, the films are imidized (cured) at 200-300°C for one hour. In production settings, an offset printer is often used for polyimide coatings. The advantages of this are better materials utilization and ability to mask off the gasket (גומי, גומי) seal area. Film thicknesses are typically 500-800 angstroms.

The cured films at this point have no preferred alignment direction—rubbing the surface of the polyimide gives it this direction. The cured films are rubbed with a velvet cloth, which is usually wrapped around a rotating drum. Alternatively, in a research or prototyping setting, load rubbing can be used. In load rubbing, a flat weight covered with velvet is drawn across the substrate at a uniform speed. The advantage of load rubbing is its reproducibility and quantitative nature. Hard rubbing is not necessary, and can lead to visible scratches (due to scattering) in the completed cell. Rub strength can be characterized by a pressure, but when a rub wheel is used, it is often useful to discuss rub strength in terms of millimeters of pile contact length. Nissan provides data using this method.

Rubbing is still one of the least understood aspects of LCD fabrication. It seems clear that there is both a mechanical and a chemical component to rubbed alignment in polyimides. Rubbing causes some grooving of the surface, which is visible by AFM, and Uchida has shown that a stamped, grooved (חריץ) epoxy will align liquid crystal. However, purely mechanical models do not accurately predict prettilts for rubbed alignment. John West has investigated purely chemical alignment by irradiation of cured polyimide films with polarized UV light (250 nm). This clearly gives a chemical anisotropy to the film and results in good alignment.

Early polyimides gave planar alignment with a small (1-3°) pretilt, but materials are now available that give prettilts up to 40°, as well as homeotropic alignment. However, obtaining reliable, reproducible prettilts in the 10-40° range is not yet possible—these materials are still in a preliminary stage. Nissan Chemical is essentially the sole supplier of these specialty polyimides, and the materials are very expensive (NOTE: although the Nissan materials are the best available, they are impractical to spincoat in a production setting—they must be printed to be cost effective). Low pretilt materials are available from numerous sources, including DuPont, and are accordingly more affordable.

Polyimide chemical structures are not always available, but some insight can be gained from prettilt studies. Some crude (גולי, מעובד ולא מעובד) classifications of the alignment mechanism can be made based on the behavior of the pretilt with increased rubbing strength. If prettilts are small, but increase with rubbing strength, the main chain structure is thought to be responsible for alignment. If prettilts are larger, and decrease with rub strength, side chains probably contribute to the pretilt. Japan Synthetic Rubber (JSR) has published numerous papers in which polyimides with known chemical structures are investigated, but little is known about most commercially available materials, especially those from Nissan.
Alignment on grooved surfaces and stamped morphology

Rubbing, tangential evaporation or shallow angle ion beam etching produce a wavy surface. It is acknowledge that any rubbing material gives good results of producing grooved surface on the substrate. However, the reproducibility of the rubbing process is not very good on substrates that are simply rubbed (as seen in the AFM photo of rubbed PI alignment layer).

A stamped morphology method has been introduced by Uchida [SID Digest, 957 (1993)] to create grooves for LC alignment. The non-rubbing alignment method has the advantage of accurate control of pretilt angle and azimuthal surface anchoring energy by changing the surface morphology. The material of the alignment layer is a bisphenol A type epoxy resin, which was added methanphenylenediamine as the hardener and xylene as the solvent. After spin coating on ITO-glass, the film was prebaked at 80°C for 10 mins. The replica was pressed on the substrate with 500 g/cm² force and kept this state until the epoxy resin is stiffened. Then cooled to room temperature before removing the replica from the substrate. In this way, the morphology of the replica is transferred to the substrate. (The photos of principle of the stamping process and morphology are shown below)

Alignment on silane and SiOₓ treated surfaces

The silanes sometimes are considered as surface active agents and sometimes as polymer forming compounds. The alkoxy silanes and chlorosilanes interact strongly with silanol groups of glass surface or hydrolysis of the silane to silanol which will further condense into a linear polysiloxane layer. Surface treatments with silanes have been effected by dipping during 5s to 1h in 1%-5% solution of the silane in water, toluene, diluted acetic in water or acetone. Water solution are only stable for a few hours. In the case of quaternary ammonium silil compounds these methods gave poor results, however, improvement was made by dipping in hot solution (75°C). Silane-treated surface give both the homogenous and homogeneous alignment, depending on the length of the alkyl chain.

The existence of hysteresis has been attributed to contamination of either the liquid or the solid, rough surfaces or absorbed surfaces film immobility.

Oblique evaporation of silicon oxide

Oblique evaporation of silicon oxide was first reported by Janning [Appl. Phys. Lett., 21, 173 (1972)]. The tilt angle achieved by this method is controlled by the evaporation angle. Goodman et al. [IEEE Trans on Electron Devices, , ED-24, 795 (1977)] proposed a columnar model based on observations by transmission electron microscope. Obliquely deposited
films have a titled columnar structure which is generated by the geometric self-shadowing of the incident atoms by those already present in the growing film. The oblique evaporation method can control the pretilt angle and the evaporated film is compatible with high temperature process.

**Langmuir-Blodgett Films**

Ikeno et al. [Japan. J. Appl. Phys., 27, 827 (1988)] demonstrated the utility of polyimide-LB films for orienting LCs without performing rubbing. Monomolecular films of amphiphilic compounds were transferred to hydrophilic substrates by dipping a cleaned glass plate into and out of a solution with constant surface pressure by means of a motor drive. Homeotropic alignment is obtained at low packing density. Using this technique it is possible to build up monolayer or multilayer functional films of organic materials. The polyamidic acid LB films for LC alignment are the most extensively studied material. The polyamidic acid LB film were prepared from alkylamine salt and then removed the alkylamine to obtain the polyamidic acid LB film.

However, twisted nematic (TN) and supertwisted nematic (STN) LCDs using these LB films have alignment defects, such as reverse twist and reverse(מכובד, ליבש כובד, רבד) tilt caused by the fact the use of LB films does not result in pretilting of liquid crystal molecules. [Ikeno et al., SID Digest, p. 45, (1988)] The quality of LB film is high in microscopic region but is poor in macroscopic areas.

**Photo-Induced Alignment of Liquid Crystals with Polarized Light**

The photosensitive polymer, poly(vinyl methoxy cinnamate) (PVMC), when crosslinked with linear polarized UV light 5 j/cm² at 320nm induces optical changes and anisotropy which force LC to align homogeneously. [M. Schadt, Japan. J. Appl. Phys., 31, 2155 (1992)] The technique has been applied to the TN cells resulting hybrid LCD with high resolution of alignment pattern. Its hybrid molecular configuration is operable in a black and white mode as well as in color.

Surface-induced alignment of nematic liquid crystals with polarized laser light was reported by Gibson et al. [Nature, 351, 49 (1991)]. The diazodiamine dye at a dye:PI weight ratio of 1:2. The chemical structure is given below. A cell using this coated glass substrate as the top plate and a second, coated with PI, as the bottom plates, separated with 11 mm spacers. Both PI layers were rubbed antiparallelly. The cell is then filled with a nematic LC ZLI-1982 and the liquid crystal is found to align along the rubbing direction. The cell is illuminated with a polarized argon ion laser (514.5 nm) with the direction of laser polarization parallel to the rubbing axis. Within the illuminated region, the molecules of LC assumed a twisted nematic structure, i.e., the LC molecules adjacent to dye-doped surface became oriented perpendicular to the rubbing direction (as shown below).
A second example of dye chromophore, tetrazaperimidine dye, was dissolved 1.8% in the liquid crystal host to form a guest-host mixture. The guest-host liquid crystal cell was then illuminated with an argon ion laser (514.5 nm) polarized along the rubbing axis. After 120 min exposure at 8 W/cm² power density, LCs within the illuminated region oriented perpendicular to the laser polarization. The LCs for the guest-host system remain aligned in the absence of the laser light and can be reoriented again by subsequent illumination. It was found that the guest-host LCs aligned this way assumed a twisted nematic structure within the illuminated region. The system may serve as a model system to probe two dimensional phase transition at interfaces.

![Figure 12](image)

**Alignments for Nematic, Smectic, and Cholesteric Liquid Crystals [T. Uchida, (1985)]**

In nematic liquid crystal cells, there are six commonly used combinations of surface alignment: Homogenous, homeotropic, twisted, hybrid, 180° STN and 270° SBE. As for smectic LC cells, there are three fundamental alignments: planar, homeotropic and focal conic. While in cholesteric LC cells, there are three corresponding alignments: planar, quasi-planar and focal conic.

**Alignment for Ferroelectric Liquid Crystals (FLC)**

One of the most important problems with FLCs is to obtain homogenous and/or bistable alignment. Because chiral smectic C phases are difficult to align with a unique molecular axis (bookshelf texture), the phase sometime shows unfavorable defect lines (zig-zag defects) of chevron (a heraldic ornament in the form of a wide inverted V-shape) structure. In order to remove zig-zag defects, uniform tilt alignment is achieved by obliquely evaporated SiO and antiparallel sealed cell. The bistable surface stabilized FLC requires weak surface anchoring. In practical, FLCs can be made to form bookshelf structures by lowering the surface energy and applying voltage.
**Spacers: Application**

Uniform cell spacing is achieved through the use of spacers. Spacers are usually glass or polystyrene spheres, or glass fibers of a precisely controlled diameter; they are usually available in half micron increments. The spacers are distributed evenly throughout the cell.

Cell thicknesses vary depending upon the type of display. The table below shows gaps for several different types of displays. Twist cell thicknesses are usually determined from phase matching conditions. STN cells in particular are extremely sensitive to thickness differences.

<table>
<thead>
<tr>
<th>Type of Display</th>
<th>Typical Cell Gap</th>
</tr>
</thead>
<tbody>
<tr>
<td>Twisted nematic</td>
<td>5-10 microns</td>
</tr>
<tr>
<td>Super twisted nematic</td>
<td>4-8 microns</td>
</tr>
<tr>
<td>PDLC</td>
<td>10-50 microns</td>
</tr>
<tr>
<td>SSCT</td>
<td>4-5 microns</td>
</tr>
<tr>
<td>PSCT</td>
<td>10-15 microns</td>
</tr>
</tbody>
</table>

**SSCT:** Surface Stabilized Cholesteric Texture. Bistable reflective displays

**PSCT:** Polymer Stabilized Cholesteric Texture (normal & reverse mode)

Choice of spacer type is dependent upon desired properties as well as display materials. Plastic spacers are necessary when plastic substrates are used. An advantage of plastic spacers is that their thermal expansion is similar to that of the liquid crystal material. This means that at higher temperatures when the liquid crystal expands and causes the cell to bow outward, the spacers will expand at the same rate, maintaining the cell gap. Glass spacers do not expand as quickly, and can float freely at higher temperatures. Glass fibers are extremely effective at maintaining cell thickness, but are not used very often in active matrix applications for fear of scratching - glass spheres are used instead.

A variety of methods are available for spacer application, though most in use are based on the same concept. The preferred method of application is to use a controlled pulse of air to disperse the spacers, which then settle onto the substrate. Many industry applicators consist of a glass box into which a fine cloud of spacers is introduced. The spacers then settle onto the substrate. This is sometimes aided by static charge, but static in the chamber (especially from triboelectric effects) can cause spacers to clump together. The LCI uses a medical device called a nebulizer to disperse the spacers. A short pulse of air (a hand-held dispenser unit works quite well for this) is applied to the nebulizer, dispersing a fine cloud of spacers from the outlet port. One advantage to dry application is that reworking is easy. A nitrogen gun can be used to remove the spacers.

Solvent assisted application can also be used. An airbrush or spray bottle can be used with a safe solvent to apply the spacers. This has the distinct disadvantage of contacting the alignment layer with the solvent. This can destroy alignment if the materials are not compatible, or cause more subtle changes like pretilt lowering (especially in high pretilt materials). In addition, substrates cannot usually be reworked, as the spacers tend to stick quite well to the substrate.
One area of interest is in the development of "sticky" spacers. If the spacers could be coated with some sort of adhesive material, adhesive curing would seal the entire area of the cell, not just the perimeter. This would eliminate the need for pressing of the cell after filling.

**Edge-Seal Applications**

Liquid crystal displays are sealed using a thin line of adhesive around the perimeter of the cell. A small port is left on one side for liquid crystal injection. For capillary filling, two opposite sides are sealed.

There is no one perfect adhesive for sealing liquid crystal displays. The adhesive must be able to maintain bond strength in very thin layers (5-10 microns), stand up to typical heat and humidity (for outdoor displays), and bond dissimilar materials well. Adhesives that bond well to both glass and polymers do not always have the desired characteristics. One solution to mask the polyimide so that the perimeter is not coated; this can be done by screen printing (difficult to coat thin enough films) or by photolithography for some polyimides. Many companies develop custom adhesives, or use combinations of commercially available brands. Adhesives for LCD’s should also be non-reactive with the liquid crystal and react close to 100% (little or no volatiles).

Edge-seal adhesives can be applied by automated or handheld dispensers, or, with smaller displays, by screen printing. Screen printing can affect the alignment layer in the cell interior through screen contact, and so is not favored for larger displays. Automated dispensers combine programming features with precise mechanical control of needle speed and adhesive flow rate; they give extremely reproducible results and allow for flexible processing. Dispenser units are available from Asymtek, Accudyne, and EFD, to name a few.

The dispensing method is usually most effective for medium viscosity materials.

UV-cure and thermal-cure adhesives are both used in the LCD industry. UV-cure adhesives are easy to work with and well-suited to dispensing application. Materials are available from Norland, Master Bond, ThreeBond, and Loctite. UV-cure materials can be formulated to bond well to glass, plastics, and metals, but do not usually have the strength or temperature stability of thermal cure materials. Thermal cure materials are usually more suited to screen printing. The LCI currently uses Norland’s NOA 68, which is very easy to work with but which has limited temperature stability.

After application of spacers and then adhesive to one substrate, the cell is assembled by placing the other substrate on top. At this point, the two substrates can be aligned if necessary, before they are pressed together for the curing process. While curing, the substrates must be pressed together so that they rest on the spacers. Thermal cure adhesives are placed in a heat press, while UV materials are pressed together so that the adhesive is exposed. One method is to place the cell on a vacuum plate and cover with a transparent, flexible material (such as Handi-Wrap) that will conform to the cell and lightly press it down onto the spacers. At this point, monochromatic light can be used to inspect cell uniformity; if nonuniform, Newton rings will be visible.

After curing, the cell will usually show interference fringes in white light. The reason for this is that stresses in the glass give it a tendency to bow, and after sealing, the two sides of the display (לדים, לאastle) outwards. These interference fringes usually form concentric rings around the cell center.
Cell Filling and Sealing

Cell filling, sometimes referred to as liquid crystal injection, is done in a vacuum chamber to minimize defects caused by trapped gases in the liquid crystal material. Capillary filling is sometimes used in a research setting because of its ease.

Degassing of the liquid crystal is extremely important. If the liquid crystal is not properly degassed, trapped gases will combine over time to produce bubbles in the display, rendering it useless in most cases. When the chamber is evacuated, trapped gases in the liquid crystal material boil out. This process is sometimes aided by ultrasonic agitation of the liquid crystal, as well as heating to the isotropic phase for filling. Although desirable, heating is a delicate process because evaporation of liquid crystal can occur. All broad range nematic liquid crystals are eutectic formed at the lowest freezing point: describes a mixture, especially an alloy, that has the lowest freezing point of all combinations or constituents, or the temperature at which this occurs) mixtures of many different pure liquid crystals, each having different vapor pressures. Care must be taken to prevent evaporation of certain components (mixtures containing PCH-32, in particular, are susceptible to this). For the same reason, ultimate vacuum pressures should not be smaller than necessary; 10-30 millitorr is usually adequate.

Once the liquid crystal material has been degassed and ultimate pressure is reached, the cell is ready to be filled. The liquid crystal is brought into contact with the display filling port, and the material begins to fill into the cell through capillary action. If the port is covered by liquid crystal, the chamber vacuum can be slowly released to speed filling--the pressure differential between the chamber and cell interior will force the material into the cell.

Filling stations are commercially available in a number of configurations, but are often custom made. The LCI uses a bell jar vacuum chamber. The liquid crystal material is placed in a trough. The unfilled cell is mounted above the trough on a movable stage, with the filling port facing the liquid crystal trough. After the chamber is pumped down and the liquid crystal degassed, the cell is lowered into contact with the liquid crystal. The disadvantage of this setup is its wastefulness. Only a fraction of the liquid crystal material is used inside the cell, the rest is wiped from the outside of the cell or lost in the transfer & pipetting from bottle to trough. A better method uses small "pitchers" of several drops of liquid crystal, which are brought into contact with the fill port.

Production filling stations are often vacuum ovens that have been modified to allow for cell filling. A long trough can be used for filling a large number of cells at once. The wick method can also be used; a wick protruding from the liquid crystal bottle is contacted to the filling port. This method eliminates the waste from pipette transfer.

After filling, the cell has once again bowed, and must be pressed down to the spacers when sealing the fill port. This is done with a bladder press. 5-10 PSI is adequate pressure to apply to the cell (pressures > 1 atm can lead to voids in the cell). After applying pressure, excess liquid crystal is wiped from the cell edge, and epoxy is applied to seal the cell.

One difficulty encountered with the filling process is flow-induced alignment. With some alignment materials, the flow of the liquid crystal during filling is enough to induce an alignment different than desired. This can be eliminated by an annealing bake after cell sealing. This heats the liquid crystal to the isotropic state, and subsequent cooling yields alignment unaffected by flow. This tends to be a bigger problem with high pretilt polyimides.
Liquid Crystal Cells Preparation
Description of Processes

Process: Alignment layer for Planar NLCs
Polyimide: HD (Hitachi Dupont) PI2555
Thinner: T9039
Mix: 1*Polyimide; 5*Thinner
Spin: 1min at 4000 RPM
Baking:
(a) Ramp to from room temperature to 250°C in 1 hour
(b) Bake at 250°C for 1 hour
(c) Down from 250°C to room temperature in 1 hour
Thickness: ~20nm (on Quartz)

Procedure:
General Requirements:

a. Cleaning chemicals: acetone, IPA, 7X detergent 5% in DI water, DI water with resistivity larger than 10M ohms, and sometimes toluene (flammable colorless liquid: a colorless liquid aromatic hydrocarbon resembling benzene, but less flammable. Use: solvent, high-octane fuel, organic synthesis. Formula: C₇H₈). The grade of chemicals to be used should be electronic grade.
b. Clean room: class 100.
c. Dress requirements: as per clean room class 100.
d. Clean room temperature: 17°C-25°C.
e. Humidity: 40-60%
f. Cleaned tools: make sure each process step has its main clean tools such as beakers, Petri dishes, and tweezers.
g. Clean room tools: filters, syringes, towels, tweezers, swabs, etc., should be appropriate for class 100 clean room with emphasis on their ability not to generate any contamination being organic, particles or ions. Tools should be stored in appropriate containers per the procedure below.
h. Storage: make sure glass and Si wafers are stored in clean, dry locations under their correct name according to batch number, date, etc. Distinguish between cleaned and dirty pieces.
i. Wafers transfer procedure: Dice Si wafers should be received from process in a labelled container together with the Wafer Transfer Form with the same label. Diced glass wafers in a labelled container should be received from the process with the Glass Run Card with the same label. You must verify that the proper label has been attached.

1. Cleaning:

The following procedure applies to non-clean pieces of glass and Si. If the pieces have undergone a cleaning procedure but were lying in a clean dry place then rinsing in IPA and acetone is usually adequate. If the pieces are coated with resist then jump to 1.7:

1.1 For quartz pieces with remnants of wax from the dicing ( 마련ת ליבריה) (otherwise jump to 1.2), you should clarify in what chemical this kind of wax dissolves. (Note: We had experienced some kind of wax that we cleaned in toluene for 45 min in ultrasound with heating and another kind that was simply dissolved in acetone without heating). After this step do not perform 1.2-1.4. Jump to 1.5.
1.2 Swab the substrates all over with cotton wool soaked in detergent.
1.3 Rinse in DI water for at least 30 sec.
1.4 Put the substrates in detergent ultrasonic bath for 30min.
1.5 Rinse in DI water for at least 30s.
1.6 Put in ultrasonic bath of DI water for 10min, three times changing the water each time. (total 30min).
1.7 Put in ultrasonic bath of acetone for 30min. (If the pieces are coated with resist start here)
1.8 Put in ultrasonic bath of isopropyl alcohol (IPA) for 30min.
1.9 Rinse with DI water and determine how clean the substrates by observing whether the water wets the substrates surfaces. If the DI water film is not uniform visually then more aggressive cleaning is required. Use the clean substrates only and leave the unclean ones aside for future re-cleaning.
1.10 Blow-dry the clean ones with clean N$_2$. Purity spec: 99.99%.
1.11 Spinning and swabbing can obtain a helpful final cleaning procedure: Put the clean substrate on the spinner centered on the chuck with the side to be coated up. Spin it at 5000RPM (ramp of 10000RPM per sec) for 1min (recipe 9) and during the spinning pour some IPA and swab it. If necessary repeat with acetone.

2. Substrates Inspection:

2.1 Look on the substrates under the microscope using 1.25x and 20x objectives for further confidence in their cleanness. The unclean ones should not be used. You may try now to re-clean with the spinner as per 1.11.
2.2 Lay the glass substrates on the optical flat under the green light and watch the surface quality (make sure the flat is clean and its bottom surface on a clean diffusive surface. If not clean then swab it with IPA carefully). If the interference fringes are straight lines then the substrate can be used, but if the curvature of the interference fringes is larger than half a fringe then do not use it. Write a note on the quality of the substrates.
2.3 Repeat 2.2 with the Si substrates but here the optical flat is on top of the Si with the bare Si side being inspected.
2.4 For long-term storage (more than a day), store the cleaned substrates in a clean dry box with silica gel sacks in it. For short-term storage (up to few hours), place the cleaned substrates in a clean Petri dish that has a clean cover until you start the next step.

3. Spin coating of polyimide:

3.1 Use the polyimide PI 2555 diluted in its thinner (1:20 by weight. The bottle is kept in the refrigerator and has magnetic stirrer in it. Put it on the stirrer (without heating) for 15 min before using. If the solution is prepared for the first time then it needs to be stirred for 10 hours before use. Replace the newly prepared bottle every 3 months.
3.2 Fill a clean syringe (if not, clean it first with DI water then with acetone and IPA and dry it with N$_2$) and attach to its end a sub-micron filter.
3.3 Put the clean substrate on the spinner centered on the chuck with the side to be coated up. Spin it at 5000RPM (ramp of 10000RPM per sec) for 1min (recipe 9) and during the spinning pour some IPA. This step is very helpful to ensure clean surface prior to the application of the polyimide. For the Si pieces spray acetone and then IPA.
3.4 If the wipers around the spinner are wet due to the IPA or acetone spraying, then preferably replace them as the vapours can arrive to the substrate and affect the PI layer (Photo Induced layer).
3.5 For the Si pieces only, the adhesion of the PI was found to be better if you heat them for 10min at 90C either in the oven or on the hot plate prior to PI application.
3.6 While the substrate on the chuck, pour the polyimide on it from the clean syringe through a sub-micron filter until it is covered. Use electronic grade filter to avoid ionic contamination.
3.7 For the Si pieces only: spin at 4000RPM (ramp of 500RPM per sc) for 1min (recipe 8). This produces a film of 20nm. For each brand new bottle check the thickness of the PI film.
3.8 For the glass pieces only: spin at 4000RPM (ramp of 3000RPM per sec) for 1 min (recipe 7).
3.9 Have a look at the backside of the substrate and clean it from excess PI with acetone and swab. Make sure the acetone does not arrive to the topside.
3.10 Lay the substrate on a clean Petri dish specifically for PI coated substrates and put in the oven at 120C for 30 min then hard bake at 245C for 1 hour then let it cool naturally with closed door. The whole process of heating the oven, maintaining it at 245C for 1 hour and cooling to say 50C should take about 3 hours.
3.11 It is possible to keep the substrates in a clean dry place for up to 72 hours before going to step 4, otherwise preferably re-heat at 120C for 30 min. Keep the coated pieces in a clean dry box with silica gel inside.

4. Rubbing:

**Using the rubbing machine:**

4.1 Blow some clean dry N\textsubscript{2} on the drum coated with the Rayon ( comunità משי) cloth.
4.2 Put the substrates on the stage of the rubbing machine and with a pen make a sign on one edge for the rubbing direction. On the Si piece you cannot make a sign with a pen, so use the contact or any other sign to make sure of the rubbing direction. Also this varies depending on the grating direction and whether the cell is TE or TM. This information can be obtained from the wafer transfer form.
4.3 Turn the motor of the rubbing wheel ON at 4 Volts.
4.4 Slowly lower the wheel and watch for the voltage measured with the voltmeter. When the voltage just starts to drop (few mVolts), stop lowering and start moving the stage towards the machine in a speed such that each substrate get rubbed 50-100 times (revolutions of the wheel). Excess pressure may cause the PI to start peeling off the surface.
4.5 Stop the voltage supply and higher the wheel so that it is possible to take the substrates out.
4.6 Take the substrates out and blow clean dry N\textsubscript{2} on them.
4.7 Cover the rubbing machine with its bag.

**Using manual rubbing:**

4.8 Put the substrates on the stage of the rubbing machine and with a pen make a sign on one edge for the rubbing direction. On the Si piece you cannot make a sign with a pen, so use the contact or any other sign to make sure of the rubbing direction. Also this varies depending on the grating direction and whether the cell is TE or TM. This information can be obtained from the wafer transfer form.
4.9 For rubbing, use the black Rayon cloth stretched on a cubical box (with a size that is easy to handle) and make sure it is replaced every 50 cells. Blow-dry N\textsubscript{2} on it before use.
4.10 Rub the substrates unidirectionally 70 times. Before rubbing blow some dry clean N\textsubscript{2} to remove dust. Do not apply any pressure, simply let the cloth feathers touch the surface. In order to make sure that the rubbing is done in the same direction you may use a rigid and straight wall in the left side to act as a guide to your hand.
4.11 Take the substrates out and blow clean dry N\textsubscript{2} on them.
4.12 Store the rubbing tools in a clean dry box with silica gel sacks inside.

5. Spacers applications and assembly:

5.1 Clean the substrates assembly holder from any dust.
5.2 Use the spacers suspension in UV61 glue inside the syringe of the dispenser.
5.3 Apply six dots, 3 along each side of the 8mm axis of the Si substrate.
5.4 Lay the Si substrate on the assembly holder. Keep the glass substrate on a clean surface.
5.5 Using clean tweezers handle with care the second substrate and lay it on top of the 1st so that the rubbing directions are anti-parallel to each other.

5.6 Press the substrates together until you start to see the interference fringes then try to adjust the pressure to get one uniform fringe. Maximum number of allowed fringes is half fringe for 1.5um cell. In order not to get deformations after removing the pressure, you should apply it on the spacers. Pressure should be the minimum adequate.

5.7 If the number of fringes is large, it means you have strong wedge or deformations originating either from the substrates themselves or from dirt. There is no point in continuing with these substrates. Try re-cleaning and replacing the spacers and if it does not improve, throw the substrates away and use new ones.

5.8 If you are satisfied with the assembly then cure the glue with the UV gun from top for 60 seconds. Make sure you wear the UV protection glasses and don’t look directly on the beam. The distance of the gun light output from the substrate should be the minimum adequate (1 cm is ok). If you have to shine UV light from larger distance then increase the exposure time according to the square of the fractional increase of the distance.

5.9 Take the assembled substrates out of the holder and seal the cell sides with UVS91 along the 8mm and part of the 5mm sides leaving inlet and outlet 1mm apertures for LC filling. Application of the glue from the sides serves also as a sealant.

5.10 Cure the UVS91 sealant using the Hg lamp for 8min when the cell is 2cm below the fibre exit. Make sure you cover the active area of the PI to block the UV from irradiating it.

5.11 Lay the optical flat on the cell surfaces under the green light and watch the quality of each surface. Write a note on the quality of the surfaces and whether the assembly has produced any deformation.

5.12 Clean and the dispensing tools with acetone and IPA, blow dry with N2 and store in a clean dry box with silica gel sacks in it.

6. LC filling:

6.1 Take the LC bottle to be used from the refrigerator few minutes before using.

6.2 Turn the oven ON to start heating up to 3 degrees above the clearing temperature (100C for E44). For the vacuum oven it takes 60min to heat up to 100C, so you may want to turn it on before the filling by 60min.

6.3 Put the assembled substrates in a clean Petri dish specifically for this purpose and make sure the oven is pre-heated to the clearing point.

6.4 With a sharp clean needle stored in a special box, take a small drop of the LC material from the bottle and put it on the edge between the two assembled substrates. The LC will start filling the gap by the capillary suction even at room temperature.

6.5 Immediately put the Petri dish with the substrates in it in the oven and pump it down to 150mBar for 2min, then slowly release the pressure to normal.

6.6 Turn the oven OFF or if you have an already prepared programme, use it so that it starts cooling at roughly a rate of 0.5C per min between 103C till 90C, then you may increase the cooling rate if you wish to 1-2C per min. This latest rate can be achieved by turning off the oven and opening the door slightly.

6.7 You can take the sample out when the temperature is below 40C.

6.8 Clean and the LC filling tools with acetone and IPA, blow dry with N2 and store in a clean dry box with silica gel sacks in it.

7. Finishing and Cell Inspection:

7.1 Clean carefully any excess LC material with a dry swab. Do not use solvent please as these can migrate inside the cell.
7.2 Visually look at the cell under the microscope. If it looks ok to you in terms of uniformity and
cleanliness, jump to the next step, otherwise there is no point to continue. Best observation is
when the polarizers are crossed and rotate the cell to see that the extinction position (dark
position) is obtained when the polarizer axis is along the rubbing direction or perpendicular to
it. Retardation colours are best observed with the rubbing direction is at 45° to the polarizer
axis.

7.3 Lay the optical flat on the cell surfaces under the green light and watch the quality of each
surface. Write a note on the quality of the surfaces and whether the LC filling has produced
any deformation.

7.4 Seal the 5mm edges of the cell with UV9S91 and cure it with the Hg lamp for 8min while
protecting the LC area from the UV. For protection use a metallic sheet or a Si sheet with
the correct size. Distance of the cell from the fiber exit should be 2cm.

7.5 Remove the PI from the contact area by scratching the PI with sharp needle.

7.6 Connect the wires using silver paste and some epoxy on top to hold them. For consistency,
the black wire to Si and the red to glass.

7.7 Clean carefully the surfaces of the substrates.

7.8 Observe retardation colours with 1.25x objective, best observed with the
rubbing direction is at 45° to the polarizer axis and the analyser crossed to the
polarizer.

7.9 Apply low frequency (up to 20Hz) 10Vpp, square wave to the cell and watch if it is flickering.

7.10 Take picture of the retardation colour map at: 0V, 5Vpp, 10Vpp at 100kHz and at 0.2-
2kHz looking for any colour nonuniformity different from that at 0V. Pick the frequency that
gives the worst colour nonuniformity.