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### Liquid Crystals

Publication details, including instructions for authors and subscription information: http://www.informaworld.com/smpp/title~content=t713926090

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**To cite this Article** Podolskyy, D. , Banji, O. and Rudquist, P.(2008) 'Simple method for accurate measurements of the cholesteric pitch using a "stripe-wedge" Grandjean-Cano cell', Liquid Crystals, 35: 7, 789 — 791 **To link to this Article: DOI:** 10.1080/02678290802175756 **URL:** http://dx.doi.org/10.1080/02678290802175756

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## Simple method for accurate measurements of the cholesteric pitch using a "stripe-wedge" Grandjean-Cano cell

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(Received 14 March 2008; final form 1 May 2008)

The classical method for measuring the inherent periodicity, or "pitch", in the order parameter in a cholesteric liquid crystal implies using a wedge cell with well-defined boundary conditions. But the error can be large due to difficulties in controlling the wedge angle,  $\alpha$ . A modified method is presented that significantly reduces the measurement error. A relief of indium tin oxide stripes on one of the plates shifts the disclination lines a distance proportional to both relief amplitude and to the wedge angle. With this "stripe–wedge Grandjean–Cano cell" the pitch can be accurately determined without a priori knowing  $\alpha$ .

Keywords: cholesteric liquid crystal; pitch measurement; stripe-wedge Grandjean-Cano cell

In cholesteric (chiral nematic) liquid crystals (CLCs) the director  $\mathbf{n}$  (the local average direction of the molecular long axes) makes a helix perpendicular to **n**. The distance corresponding to a full rotation of the director is called the pitch, p. The value of p can be determined in several ways (1-3), some of which are relatively simple but lacking in accuracy. Small pitch materials  $(p \sim \lambda)$ , where  $\lambda$  is the wavelength of light) constitute one-dimensional (1D) photonic bandgap materials and p can be calculated from  $p = \lambda_r/n$ , where  $\lambda_{\rm r}$  is the centre wavelength of selective reflection of light incident along the helix axis, and *n* is the average refractive index of the CLC (4). For accurate determination of p we need to know the value of n. In long-pitch materials, p can be estimated from the diffraction of light incident perpendicular to the helix axis or directly from the fingerprint structure in polarising microscopy. In the fingerprint structure, however, p often deviates from its bulk value due to surface action (5). An alternative method for determination of the helical pitch for long-pitch values was proposed by Suh et al. using circular rubbing on one of the substrates (6).

The classical, most straight-forward and common method for determination of p is, however, the so-called Grandjean–Cano wedge method (1, 2). It is illustrated in Figure 1.

The two cell plates are assembled to form a wedge with an opening angle  $\alpha$ . The boundary conditions make the CLC helix orient perpendicular to the cell walls. When the cell gap increases continuously along the wedge, the integer number of half pitches increases in a discontinuous way through disclination lines. The value of the pitch is calculated from the distance *L* between the disclination lines and the angle of the wedge  $\alpha$  as

$$p \approx 2L\alpha.$$
 (1)

This direct method is simple and is applicable for a wide range of pitch values by using wedges with different values of  $\alpha$ . However, difficulties in controlling or measuring  $\alpha$  severely limit the accuracy of the pitch measurements. In hand-made wedge cells, the error in the estimation of  $\alpha \approx (t_2 - t_1)/T$  can be larger than  $\pm 20\%$ . Here  $t_1$  and  $t_2$  are the cell thickness at the beginning and the end of the wedge and *T* is the length of the wedge, as shown in Figure 1. For instance, with  $t_2 - t_1 = 10 \pm 2\,\mu\text{m}$  and  $T = 10 \pm 0.2\,\text{mm}$ ,  $\alpha$  equals  $1 \pm 0.20\,\text{mrad}$ . Moreover, the angle  $\alpha$  can vary along the cell, which decreases the accuracy of the measurement even further.

By using a spherical lens with a well-defined radius as one of the substrates, the angle  $\alpha$  becomes a function of the distance *R* from the point of smallest gap between the flat substrate and the cell. The disclination lines now form concentric circles around this point with the mutual distances,  $R_{i+1}-R_i$ , between adjacent disclinations decreasing for increasing *R*. With the radius of the lens  $r_{\text{lens}}$  the pitch is given by  $p = (R_{i+1}^2 - R_i^2)/r_{\text{lens}}$  (2, 7). However, this is a cumbersome method, involving putting an alignment layer on the curved surface of the lens and creating an oriented sample (8).

In this paper we report a method in which we circumvent the problem of controlling  $\alpha$  in the

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Figure 1. Schematic illustration of the Grandjean–Cano wedge technique. The disclination lines are perpendicular to the plane of the paper. The pitch,  $p=2L\alpha$ , where L is the distance between two disclination lines and  $\alpha$  is the wedge angle.

Grandjean–Cano wedge. We have created a wellcontrolled surface relief in the form of indium tin oxide (ITO) stripes with known thickness along the wedge on one of the plates of the wedge (see Figure 2). The thickness of the cell still varies continuously along the opening direction of the wedge but, importantly, discontinuously in the perpendicular direction.

The sharp variation in thickness caused by the surface relief shifts the disclination lines a distance *l*, proportional to the relief amplitude *d* and to the opening angle  $\alpha$ , i.e.  $\alpha = dll$  (see Figures 2 and 3). Using equation (1) the pitch can easily be determined through

$$p = 2Ld/l. \tag{2}$$

From equation (2) it can be seen that with this "stripe-wedge Grandjean-Cano cell" the pitch can



Figure 2. Illustration of the stripe–wedge Grandjean–Cano cell. The shift of the disclination lines, l, due to the surface relief amplitude, d, gives p=2Ld/l, where L is the distance between successive disclination lines.



Figure 3. Microphotograph of a stripe–wedge Grandjean– Cano cell filled with TI827. The wedge thickness increases from left to right. The disclinations (vertical) are perpendicular to the cell thickness gradient and at the stripe edge the disclinations shift the distance *l*. The diameter of the spacers is  $9 \,\mu\text{m}$ .

be accurately determined without a priori knowing the wedge angle  $\alpha$ . The distances *l* and *L* are easily measured by means of optical microscopy (Figure 3), and the amplitude of the surface relief, *d*, can be accurately measured by means of atomic force microscopy (AFM) or alpha-step measurements. Importantly, the accuracy in the ratio *dll* in equation (2) is much higher than for an estimated value of  $\alpha$  in a conventional wedge. Hence the stripe–wedge Grandjean–Cano cell significantly improves the accuracy of pitch determination compared to the conventional Grandjean–Cano method, as will be discussed below.

We have manufactured stripe-wedge Grandjean-Cano cells in-house at the Chalmers Nanofabrication Laboratory. We started from pairs of  $3'' \times 3''$  ITOcoated glass plates (conventional display glass) on which the surface relief was created on one of the plates by means of photolithography. As alignment layer we used the polyimide 2610 (Pyralin), which was spin-coated onto both plates, cured and subsequently rubbed using a commercial rubbing machine (LC-Tec Automation). The amplitude of the surface relief was determined by AFM to be 85 nm with a mean surface roughness of less than 1 nm. The substrates were glued together using a UV-curing glue (NOA 68, Norland Adhesives), which was dispensed onto the bottom plate using an automatic glue dispenser (Asymtek). The thickness gradient in each cell was assured by putting polymeric spacer balls with a diameter of 9 µm in selected parts of the glue pattern, as illustrated in Figure 4. Each assembly of  $3'' \times 3''$ plates was cut into 20 stripe-wedge cells. This manufacturing method allows for production of large quantities of stripe-wedge cells and by the choice of the spacer diameter we can optimise the cells for measurement of different ranges of pitch values.



Figure 4. Photograph of a  $3'' \times 3''$  assembly before cutting into 20 stripe–wedge cells. Each cell is  $12.5 \times 12.5$  mm (indicated with red boxes). The wedges are formed by using different diameters of the spacers in the two glue lines cementing the cells. Any remaining curvature of the upper plate in the final cells can be neglected since *l* and *L* are measured locally in the stripe–wedge cell.

Figure 3 shows a close up of a stripe-wedge cell filled with a cholesteric mixture TI827 (Merck). On either side of the thickness step, the disclination lines are slightly curved. This is not due to thickness gradients; the step is indeed sharp. The disclination lines are in fact continuous across the step and become parallel to the surface relief at the step. In the region around the step, in which the disclination lines are curved, the cholesteric is further elastically deformed. L and l are therefore measured away from this "relaxation" region, where the disclination lines are straight. With the horizontal edge of the image being 170 mm we measure L=111+1 mm and  $l \approx 41 \pm 1$  mm, respectively. From the AFM measurements we know that the surface of the polyimidecoated ITO is very flat and the uncertainty in relief amplitude should be due to global variations in ITO layer thickness. Repeated AFM measurements on several samples indicate that the spread in relief amplitude is less than 2 nm, i.e. d=85+2 nm. Adding the relative errors  $(\delta L/L + \delta l/l + \delta d/d = 1/111 + 1/41 + 2/85)$ we get a total error of 5.6% from this single measurement and  $p=460\pm26\,\mathrm{nm}$ . With  $d=85\pm5\,\mathrm{nm}$ the maximum error would still be less than 9%. Furthermore, the errors can be further reduced

through averaging over many measurements of the ratio L/l in the same cell. The resulting error of a few percent in the estimated pitch value using the stripe-wedge Grandjean–Cano method is thus significantly smaller than the possible large errors related to the difficulties in determination of  $\alpha$  using a conventional Grandjean–Cano wedge.

In conclusion, we have proposed and demonstrated a modified Grandjean-Cano method for the simple and accurate determination of the pitch of cholesteric liquid crystals. The wedge opening angle does not have to be accurately controlled and the method gives a reasonable accuracy in contrast to the conventional Grandjean-Cano wedge. The remaining inaccuracy in the pitch value is mainly related to the precision in the determination of the relief amplitude. In the example given here, we have assumed +2 nm and +5 nm from AFM measurements and the surface roughness of 1 nm. By using a crystal surface, e.g. Si wafers and even epitaxial growth and lithography of the relief material the precision in d will be further increased. A surface roughness of a few nanometres should, however, have a small influence on where the disclination lines form. Instead it is the average local distance between the two cell surfaces that is important. In fact, local variations in the glass thickness should have a larger influence than the surface roughness. But as the stripe-wedge cell indirectly gives a local value of the wedge angle,  $\alpha$ , changes in average glass thickness over distances of the order L in the cell should be small facilitating accurate determination of the cholesteric pitch.

#### Acknowledgements

The authors would like to acknowledge Dr P. Jägemalm and Dr Å. Haglund, for performing the AFM measurements. Part of this work was supported by The Swedish Foundation for Strategic Research, Dnr 2005/0257.

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