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# Molecular Crystals and Liquid Crystals Incorporating Nonlinear Optics

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## Scanning Conoscopy: a Novel Method for Studying Birefringent Samples

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# Scanning Conoscopy: a Novel Method for Studying Birefringent Samples

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We describe a novel conoscopic method for the accurate determination of the optic axis and the thickness of birefringent samples such as liquid crystals. The method consists of synthesizing the entire effective range of the angle of incidence by rotating the sample about a narrow incident beam, thus producing a very large numerical aperture for the incident beam. Furthermore, the sample observation is made locally over a small area and unlike the conventional microscope conoscopy, the proposed scanning technique offers an additional advantage in terms of an extremely high working distance. In the case of a liquid crystal application, this helps us to add any auxiliary equipment around the sample under observation.

#### 1. INTRODUCTION

Microscope conoscopy has proven to be an efficient technique for studying birefringent materials.<sup>1</sup> In this technique the quantitative measurement of the crystal parameters can be obtained by suitably analysing the conoscopic interferogram and the precision of this measurement is determined by the numerical aperture of the microscope objective. High precision measurements are possible, however, by using immersion optics but this makes the technique cumbersome and less suitable for applications requiring auxiliary equipments such as a heating stage around the sample. Furthermore, the fact that a wide sample area is analysed at the same time, it renders the technique

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still less interesting for the purpose of studying small monodomains of crystals. In this paper, we describe a novel simple approach for conoscopic observations allowing accurate measurements of crystal parameters. The method consists of scanning the effective range of the angle of incidence by rotating the sample about the fixed narrow incident beam. In other words the numerical aperture of the incident beam is synthesized and its effective value can reach as high as unity. The minimum width of the analysed area under observation has been estimated to be approximately of the order of the sample thickness. In addition to the inherent advantage in terms of high sensitivity offered by a large numerical aperture, a wide space around the sample becomes available for adding any auxiliary equipment. These two features make the proposed scanning technique very versatile for studying the orientational behavior of liquid crystals under variable surrounding conditions including the presence of an external electric or a magnetic field.

#### 2. THEORETICAL

The conoscopic observation of anisotropic materials consists of studying the interference between the ordinary and the extraordinary waves emerging from the sample over a range of the angle of incidence. In the proposed scanning configuration this range is synthesized by rotating the sample illuminated by a fixed narrow incident laser beam. Figure 1 shows the experimental set up, where a spatially filtered beam from a low power He-Ne laser is focused onto the sample with a microscope objective having a low numerical aperture.

The input beam is perpendicular to the axis of rotation and intersects it at a point which will henceforth be referred to as the center of rotation. It is essential that the axis of rotation is contained in the input face of the sample in order to ensure the same and the smallest

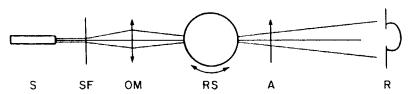


FIGURE 1 Experimental set-up for scanning conoscopy. S: Laser; SF: spatial filter; OM: Microscope objective; RS: Rotating stage; BS: birefringent sample; A: analyser; R: radiometer.

observation area for all angles of incidence. Such an alignment is achieved by mounting the sample on an assembly of motorized micropositioning stages as detailed in the next section. Assuming that the appropriate adjustments have been made, the angle of incidence is then given by the angle of rotation of the stage. The output beam is analysed with a polariser which is crossed with respect to the direction of the input beam polarisation. The intensity of the output beam is measured with a radiometer and plotted on an XY recorder as a function of the angle of incidence.

For a given birefringent plate of thickness d, having its optic axis lying in the plane of incidence, the output intensity curve can be written as a function of the angle of incidence by the following expression:<sup>2</sup>

$$I(i,\theta) = I_0 \int_{\alpha} \sin^2 \left(\frac{\Delta \phi}{2}\right) di, \qquad (1)$$

where

$$\Delta \phi = \frac{2\pi}{\lambda} d[n(i,\theta) \cos r(i,\theta) - n_0 \cos r_0(i)]. \tag{2}$$

In Equations (1) and (2), i and r are respectively the angles of incidence and refraction for the sample,  $\theta$  is the angle of inclination of the optic axis,  $\alpha$  is the radiometer aperture and the other symbols have their usual meanings. If the refractive indices of the sample are known, the comparision between the experimental curves  $I = f(i,\theta)$  obtained from the scanning conoscopy and the corresponding theoretical curve computed from the Equation 1 would lead to the determination of the sample thickness and optic axis direction.

For a given angle of incidence, the order of interference is determined by the thickness d and the inclination of the optic axis  $\theta$ . The zeroth order fringe does not depend on the sample thickness and is given by the condition when the illuminating beam in the interior of the sample (refracted beam) is parallel to the optic axis. In the vicinity of the zeroth order fringe, the phase retardation  $\Delta \phi$  varies slowly with respect to  $(i-\theta)$  and the width of this fringe is always larger than the others. This feature enables us to identify and label without any ambiguity the order of the fringes in the interferogram. Thus, the position of the zeroth order fringe on the experimental curve readily gives us the direction of the optic axis. The sample thickness can now be calculated from the Equations 1 and 2 by using the

experimental value of the fringe orders corresponding to the extreme values of the angle of incidence.

#### 3. EXPERIMENTAL

As shown in Figure 2, the sample is aligned with respect to the incident beam by mounting the stationary part of the rotating stage on a translation stage  $(T_1)$  lying in the plane  $(X_1Y_1)$ . It is used to bring the rotation axis, lying along the  $Z_1$  direction, to intersect the input beam at its focal point. A second translation stage  $(T_2)$  is mounted on the rotating part. The sample is then mounted on the stage  $(T_2)$  in such a manner that its input face is perpendicular to the  $(X_2Y_2)$  plane. The translation stage  $(T_2)$  is used to bring the input face of the crystal to contain the axis of rotation. In this way the center of rotation and the point of incidence on the sample become coincident.

As it can be seen from Figure 3, any misalignment of  $T_1$  and  $T_2$  will change the observation area over the crystal. While studying a sample with parallel faces, such a misalignment would not alter the numerical value of the thickness and the optic axis direction if the

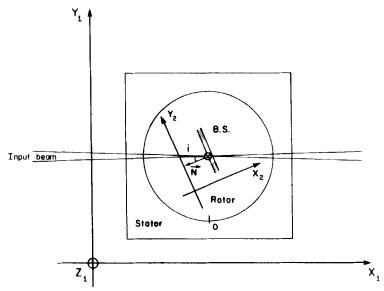


FIGURE 2 Multiple axis translation and rotation assembly for the sample. BS: bi-refringent sample; N: normal; i: angle of incidence; X Rotation axis of the rotating stage.

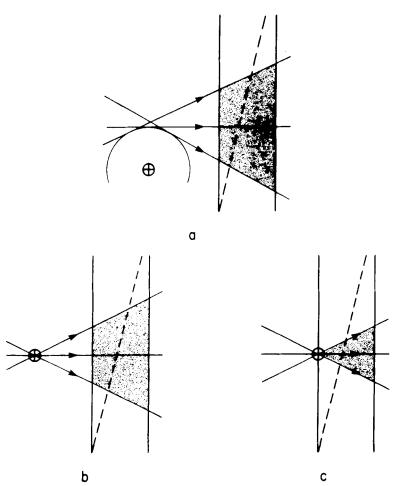


FIGURE 3 Alignment of the sample. a) Showing input face centre of rotation (X) and input beam misaligned. b) Showing centre of rotation (X) and input beam aligned and input face misaligned. c) Showing the correct alignment.

observed area is a monocrystal. Thus in the case of a monocrystal plate, a visual inspection of the alignment is adequate. However in the case of a liquid crystal sample whose optical axis direction varies rapidly in the bulk, it is necessary to align the sample more precisely for the purpose of studying its smallest possible area. This can be done by using the sample in the form of a wedge. Its position is adjusted by using  $(T_2)$  until an exact match between the theoretical and experimental output intensity curves is obtained. The following

expression for the phase retardation is used for computing the theoretical output intensity curve:

$$\Delta \phi = \frac{2\pi}{\lambda} d \left[ \frac{n \cos r \cos i}{1 - \tan r \cdot \tan \delta} - \frac{n_0 \cos r_0 - \cos i}{1 - \tan r_0 \cdot \tan \delta} \right], \quad (3)$$

where  $\delta$  is the apex angle of the wedge. For small values of  $\delta$  ( $\sim$ 1°), the curve  $I = f(i,\theta)$  is quasi-symmetrical with respect to the angle  $(i-\theta)$  when the point of incidence and the centre of rotation are coincident. As these two points move away from each other, the curve becomes more asymmetrical.

#### 4. DISCUSSION

One of the attractive features of the microscope conoscopy consists of displaying the fringe pattern simultaneously over the entire range of angles and planes of incidence of the illuminating beam. For a given sample, the precision of measurement is determined by the total number of fringes in the field of view of the microscope. For this purpose, it is desirable that the microscope objective has a wide numerical aperture. Typically, angles of incidence in the range  $\pm 40^{\circ}$ would require an objective having a numerical aperture of 0.65 and an associated working distance of about 0.8 mm. In this case, it is practically impossible to insert an auxiliary gadget like a heating stage around the liquid crystal sample. Assuming the minimum required working distance for this purpose to be 15 mm, the available numerical aperture of the microscope objective drops down to about 0.2. Unlike the above example, the angles of incidence of the illuminating beam in the present method can attain values up to  $\pm 90^{\circ}$ which correspond to a numerical aperture of 1. The fact that the effective aperture is synthesized by rotating the sample around a narrow pencil of light, it imposes no limitations on the working distance. In practice, the working distance is fixed by optimizing the parameters of the laser beam assembly. In our experiment, this distance has been equal to 25 cm.

In microscope conoscopy all angles and planes of incidence are examined simultaneously. On the contrary, in the scanning conoscopy, the experimental output intensity versus the angle of incidence curve corresponds to a diametric section of the microscope field of view as shown in Figure 4. A different orientation of this section in the field can be selected by rotating the sample in the  $Y_2Z_2$  plane as given by the Figure 2.

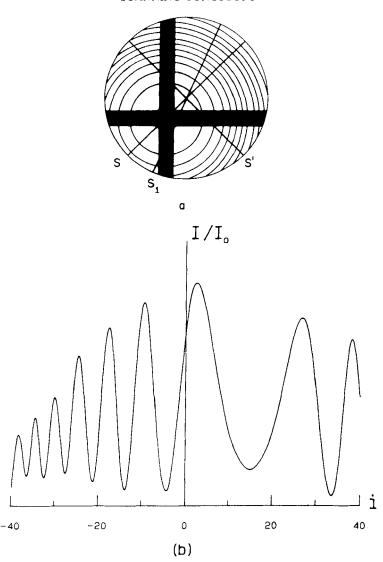


FIGURE 4 Comparison between microscopy and scanning conoscopy. a) Field of view in microscopy conoscopy. b) Scanning conoscopy record corresponding to the cross-section S.

A quantitative determination of the crystal parameters requires an image analysis operation involving radiometric measurements of the field of view in microscope conoscopy. Also an intermediate step consisting of making a photographic recording of the microscope field of view is necessary to produce the data. In scanning conoscopy this

information is provided directly by the output intensity curve and in certain cases, the interpretation of such a curve is comparatively simpler than the visual inspection of the microscope conoscopy photographs. As an example we consider the case involving the study of orientational behavior of a homeotropic liquid crystal cell. Above the Freedericksz transition threshold, a molecular reorientation of liquid crystals under the influence of an external electric field gives rise to a rotation of the sample optic axis.3 This reorientation can occur in two different ways as shown in Figure 5. Figure 5(a) shows schematically what has been called as the one-fold molecular reorientation. This produces a simple shift in the fringe pattern parallel to the fringes as shown in Figure 6(a). The set of curves shown in the Figure 6(a) corresponds to various values of the voltage applied to the liquid crystal sample. The threshold value of the Freedericksz transition can be determined from these curves by suitably plotting the position of a given fringe order against the applied voltage. The abscissa of the sharp bend in this curve corresponds to the threshold value of the Freedericksz transition. Similarly the two-fold molecular reorientation case of the Figure 5(b) produces an output intensity curve as shown in the Figure 6(b). It can be seen that the output intensity curve in the Figure 6(b) is marked by the presence of comparatively lower intensity maxima. There might exist an ambiguity in the interpretation of the experimental output intensity curve. The Figure 6(b) can also be considered to represent a misaligned one-fold reorientation of crystals where the optic axis has become out of the plane of incidence. This corresponds to the case where the scanning has been made along the crystal cross-section S' as shown in the Figure 4(a). The distinction between these two cases can be made by comparing the symmetry of the output intensity curves recorded in two orthogonal cross-sections of the field of view.

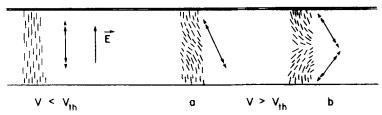


FIGURE 5 Electric-field induced molecular reorientation of homeotropic nematic film. E: electric field;  $V_{\rm th}$ : threshold of the Freedericksz transition. The double arrows refer to the optic axis.

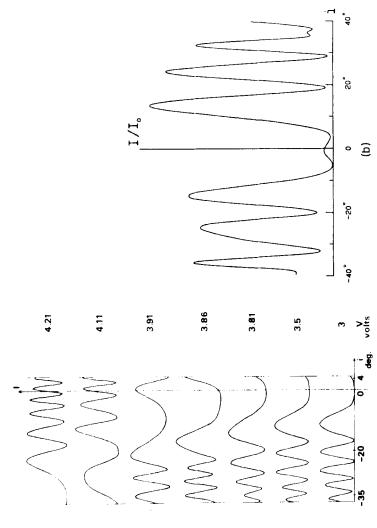


FIGURE 6 Scanning conoscopy recordings corresponding to: (a) molecular reorientation as shown in Figure (5a) and (b) molecular reorientation as shown in Figure (5b).

In the two-fold reorientation case producing the experimental curve shown in Figure 6(b), the plane of incidence containing the two optic axes, is a plane of symmetry and hence the scanning conoscopy curve in the perpendicular cross-section is symmetrical with respect to the angle of incidence. Such a symmetry does not exist (cross-section S, Figure 4) in the one-fold reorientation case where the optic axis is tilted out of the plane of incidence. Furthermore, the scanning conoscopy has been found to detect optic axis tilt angles as low as 1°. This feature makes this technique extremely useful for thin samples and for samples with small tilt angles.

Finally, it can be noticed from the Figures 4(b) and 6 that the output intensity curves do not vary between zero and a fixed maximum. This effect becomes more prominent for high angles of incidence. The corresponding loss in the fringe visibility has been attributed primarily to the polarization dependence of the light reflected at the air-glass interfaces. Consequently, a correction needs to be applied to the formula describing the transmitted output intensity. Furthermore, the loss in the fringe visibility can also be caused by the finite size of the detector which is possibly too large in comparison with the interfringe spacings corresponding to high angles of incidence.

### 5. CONCLUSION

We have described a simple apparatus for the conoscopic study of birefringent materials. In contrast to the relatively qualitative techniques such as microscope conoscopy, the scanning conoscopy provides directly quantitative information for an accurate determination of both the optic axis and the thickness of the sample. The observation is made locally over a very small area of the sample using an extremely narrow beam of light. This makes available ample space around the sample for inserting any additional equipment. The method is, thus, highly suitable for measurements on liquid crystal samples. In a forthcoming paper, we shall use this technique for measuring the anchoring energy of weakly anchored nematic cells. Finally, the proposed technique of scanning conoscopy supplements the method of microscope conoscopy and the two are not in direct competition with each other. Furthermore, this method may be considered to be complementary to coherent orthoscopic microscopy due to Peri et al<sup>4</sup> and the method by Cladis<sup>5</sup> for studying twisted materials.

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