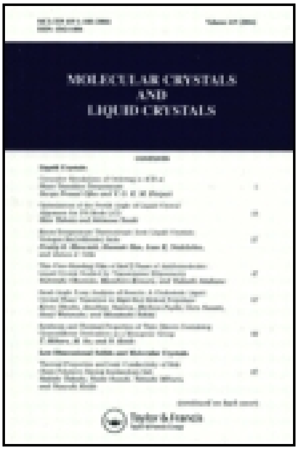

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Method for Determining Material Constants of Nematic Liquid Crystals by Use of Wedge Cells

Jerzy Kędzierski [a] , Zbigniew Raszewski [a] , Jolanta Rutkowska [a] , Marek Andrzej Kojdecki [b] , Ludwika Lipińska [c] , Emilia Miszczyk [d] & Jerzy Borycki [e]

[a] Institute of Applied Physics, Military University of Technology, Warsaw, Poland

[b] Institute of Mathematics and Cryptology, Military University of Technology, Warsaw, Poland

[c] Institute of Electronic Materials Technology, Warsaw, Poland

[d] Institute of Physics, Radom University of Technology, Poland

[e] Faculty of Chemistry, Radom University of Technology, Poland

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METHOD FOR DETERMINING MATERIAL CONSTANTS OF NEMATIC LIQUID CRYSTALS BY USE OF WEDGE CELLS

Jerzy Kędzierski, Zbigniew Raszewski, and Jolanta Rutkowska*
Institute of Applied Physics, Military University of Technology, 00-908 Warsaw, Poland

Marek Andrzej Kojdecki
Institute of Mathematics and Cryptology, Military University of Technology, 00-908 Warsaw, Poland

Ludwika Lipińska
Institute of Electronic Materials Technology, 01-919 Warsaw, Poland

Emilia Miszczyk
Institute of Physics, Radom University of Technology, 26-600 Radom, Poland

Jerzy Borycki
Faculty of Chemistry, Radom University of Technology, 26-600 Radom, Poland

A wedge cell of the wedge angle of order of few milliradians was used to measure the phase shift between ordinary and extraordinary rays of transmitted light, for the Fréedericksz transition induced by a magnetic or an electric field. A nematic liquid crystal, PCB, filling the cell, was of the planar alignment enforced by the treatment of the flat boundary plates. A polyimide, MK8–poly (amic acid), was used as aligning substance. A system of interference fringes appeared in the cell placed in normally incident light between the analyser and the polariser crossed. In the neighbourhood of each fringe the cell can be considered as a flat-parallel one and hence it is equivalent to a system of flat cells of different precisely determined thickness. The dependence of the phase shift on applied external fields was measured for several interference fringes. The nematic liquid crystal material parameters (the splay and bend elastic

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* Corresponding author. E-mail: jkedzierski@wat.edu.pl

constants, the anisotropy of diamagnetic susceptibility and the boundary tilt angle as a function of the torque) were determined by fitting the calculated characteristics to the measured ones.

Keywords: 4′-pentyl-4-cyanobiphenyl (PCB); inverse problem; nematics elastic constants; nematics magnetic susceptibility; nematics parameter identification; polyimides

INTRODUCTION

The problem of determining the values of material constants for nematic liquid crystals is very important for both the theory and the applications of them. The verification of the level of adequacy of theory as well as realistic computer simulation of physical experiments is possible only if the proper magnitudes of material constants are known. Some of them, like the Frank elastic constants and the anisotropy of diamagnetic susceptibility, can not be measured immediately and must be determined as solutions of corresponding inverse problems, in which the characterisation of the coupling between a nematics and a substrate should be necessarily taken into account. Measurements are made usually with the use of standard flat-parallel nematic cells. The measured quantity, monitoring both the director field inside the cell and the material constants, is the effective electric permittivity or the optical response of a nematics layer. Due to specific feature of inverse problems, particularly possible instability of solutions with respect to errors in data, some reasonable excess of input information is required to obtain the reliable magnitudes of material constants. It can be provided by measuring different characteristics (e.g. influenced by several combinations of an electric and a magnetic field) for different cells. The wedge cell gives the advantage since it can be treated as equivalent to a system of flat cells with exactly the same coating surface treatment and precisely defined thickness.

WEDGE CELL FOR MEASURING OPTICAL CHARACTERISTICS OF NEMATICS

A wedge nematics cell was made of glass plates (of size $22 \times 35\,\mathrm{mm}$), coated with indium-tin oxide electrodes and orienting polyimide layers [1]. The plates were glued without a spacer along an edge and with a spacer (of thickness about $200\,\mu\mathrm{m}$) along the opposite one. The orientation of the nematics molecules enforced by the substrates was parallel to the boundaries and to the wedge edge. The cell was placed in a thermostatic stage between the polariser and the analyser crossed in the measurement system, consisting of a He-Ne laser and a microscope with a photodetector,

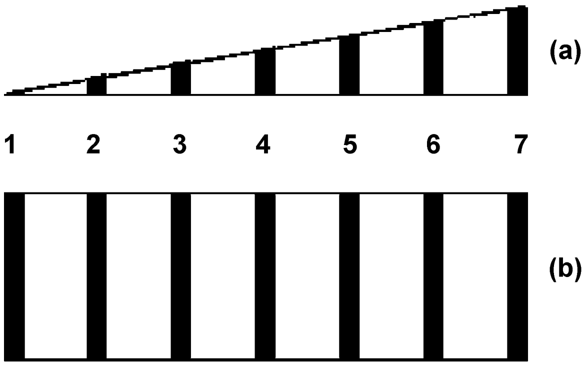

FIGURE 1 System of interference fringes of normally incident light in a wedge cell, filled with a nematics of planar alignment, in the absence of external fields. The subsequent fringes correspond to the subsequent orders j (displayed in the figure) of interference minimum; the corresponding cell thickness d, the difference of optical paths Δ and the phase shift Φ are related by the following formulae: $\Phi_j = \frac{2\pi}{\lambda} \cdot \Delta_j = 2\pi \cdot j,\ \Delta_j = \Delta n \cdot d_j = j \cdot \lambda,\ d_j = \frac{j \cdot \lambda}{\Delta n}\ \Delta n = n_e - n_0$. (a) denotes the side view and (b) denotes the top view.

and between pole pieces of an electromagnet. In the normally incident light a system of interference fringes appeared, like shown in schematic Figure 1. In the small neighbourhood of each fringe position a wedge cell can be treated as the planar one (like sketched in Figure 2) due to very small wedge angle. The intensity of normally incident light transmitted through

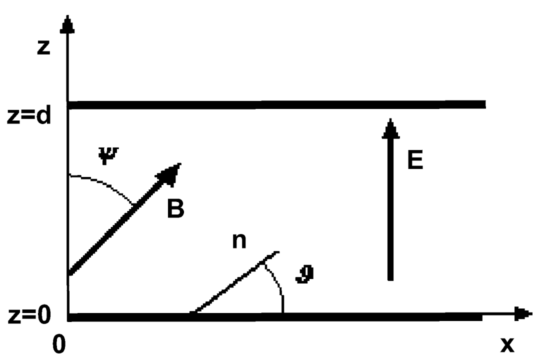

FIGURE 2 Sketch of the experimental configuration of an equivalent flat nematics cell in external fields. The director, Ox and Oz axes and external (magnetic or electric) field vectors are all in the same plane.

the cell in the positions of selected interference fringes was recorded as a function of an applied voltage and a magnetic field. In such experiment a wedge cell is equivalent to a system of planar cells of different thickness.

STUDIED MATERIALS

The polyimide MK8 [2] was used as an aligning substance [3]. MK8 was poly(amic acid) produced by reacting 4,4′-oxydianiline with 4,4′-oxydiphthalic anhydride. The glass plates with indium-tin oxide electrodes were spin-coated with the poly(amic acid) solution in dimethylformamide and dried. The thermal imidisation process was then carried out at the temperature 250°C for 4 hours. Finally the plates were parallel rubbed in the direction parallel to the wedge edge.

The cell was filled with 4′-pentyl-4-cyanobipfenyl (5CB or PCB). Its electric permittivities were measured in a specially constructed double flat condenser built of three silver electrodes, each of area 6.25 cm^2, and filled with 10 cm^3 of liquid crystal [4], by using a sinusoidal voltage of frequency 1592 Hz. The temperature was stabilised with the accuracy within 0.2°C in the whole range and the relative error of measurements was about 0.1%.

The extraordinary and ordinary refraction indices of PCB for the He-Ne laser red light (of wavelength 0.6328 μm) were measured by an Abbé refractometer, equipped with a water jacket to control the temperature. The surface of the main prism was coated with a solution of lecithin in methanol to align the director homeotropically. The extraordinary refractive index was measured with an eye-piece polariser parallel to the director and the ordinary refraction index was measured with a polariser perpendicular to the director. The temperature was stabilised with the accuracy within 0.3°C in the whole range and the error of measurements in the nematic state was about 0.0005.

The temperature of phase transition between nematic and isotropic state was found to be equal to 34.5°C.

EQUIVALENT FLAT-PARALLEL CELL

In the small neighbourhood of each fringe, observed in the experiment, the wedge cell was described as the flat-parallel cell (Fig. 2). An electric field, induced by a voltage, and a magnetic field acted in the plane of the free alignment. In this way all deformations were planar. The one-dimensional approximation was used i.e. physical quantities were assumed as depending only on z e.g.:$\vec{n}(z) = (\cos\vartheta(z), 0, \sin\vartheta(z))$, $\vec{E}(z) = (0, 0, E(z))$, $\vec{B}(z) = (B\sin\psi, 0, B\cos\psi)$.

DESCRIPTION OF PHENOMENA

The flat nematics cell of thickness d is treated with good accuracy as the layer infinitely extended in two dimensions (in Oxy plane) and placed between two parallel planes ($z = 0$ and $z = d$). Moreover let the boundaries cause a homogeneous stationary state of the layer (described by a constant director field) and let the external fields act in the same plane Oxz, as in Figure 2. The static deformations of such cell, caused by constant fields, an electric one $\vec{E} = \vec{E}(z) = (0, 0, E(z))$ induced by a constant or low-frequency voltage U applied to the layer boundaries (i.e. the cell cover electrodes), and a magnetic field $\vec{B} = \vec{B}(z) = (B\sin\psi, 0, B\cos\psi)$, are planar and can be described in a one-dimensional approximation by the planar director field $\vec{n} = \vec{n}(z) = (\cos\vartheta(z), 0, \sin\vartheta(z))$. Analysing the functional of free elastic energy of the nematics layer influenced by external fields, corresponding to such configuration, one obtains [8,9,16,17] the Euler equation for it in the following form of the system of two equations:

$$\begin{aligned} &(K_{11}\cos^2\vartheta + K_{33}\sin^2\vartheta)\vartheta'' + (K_{33} - K_{11})\cdot\sin\vartheta\cdot\cos\vartheta\cdot\vartheta'^2 \\ &\quad + \frac{\varepsilon_0\varepsilon_a\varepsilon_e^2 U^2 \sin\vartheta\cos\vartheta}{d^2(\varepsilon_\perp\cos^2\vartheta + \varepsilon_{||}\sin^2\vartheta)^2} + \frac{\chi_a B^2}{\mu_0}\sin(\vartheta+\psi)\cos(\vartheta+\psi) = 0 \\ &\varepsilon_e = \left\{\frac{1}{d}\int_0^d \frac{1}{[\varepsilon_\perp\cos^2\vartheta(z) + \varepsilon_{||}\sin^2\vartheta(z)]}dz\right\}^{-1} \end{aligned} \quad (1)$$

where: K_{11}, K_{33} are the splay and bend Frank elastic constants, $\varepsilon_\perp, \varepsilon_{||}$ are the electric permittivities (measured perpendicularly and parallel to the long axis of molecules), $\varepsilon_a = \varepsilon_{||} - \varepsilon_\perp$, χ_a is the anisotropy of diamagnetic susceptibility, ε_e is the effective electric permittivity of the layer, ψ is the angle between the direction of a magnetic field and the layer normal and the elastic free energy functional is assumed in the form with three Frank elastic constants [5–8]. The phase shift between ordinary and extraordinary rays of light, incident normally on the cell, changes in accordance with a nematics state and a resulting value of the effective refractive index of the layer n_{eff}:

$$\begin{aligned} \varphi &= \frac{2\pi}{\lambda}d\Delta n_{eff} = \frac{2\pi}{\lambda}d(n_{eff} - n_o) \\ &= \frac{2\pi}{\lambda}d\left[\frac{1}{d}\int_0^d \frac{n_e\cdot n_o}{\sqrt{n_e^2\cdot\sin^2\vartheta(z) + n_o^2\cdot\cos^2\vartheta(z)}}dz - n_o\right], \end{aligned} \quad (2)$$

In general the weak anchoring at the layer boundaries should be assumed. It can be characterised by a suitable boundary condition for ϑ.

The elastic torque density (per unit area of the layer boundary), transmitted from the bulk, can be calculated [10–12,16,17] for function ϑ, satisfying Eq. (1), as

$$T_b = \int_0^d \left[(K_{11} - K_{33}) \cdot \sin\vartheta(z) \cdot \cos\vartheta(z) \cdot \vartheta'(z)^2\right] dz + \int_0^d \left[\frac{\varepsilon_0 \varepsilon_a \varepsilon_e^2 U^2 \sin\vartheta(z)\cos\vartheta(z)}{d^2[\varepsilon_\perp \cos^2\vartheta(z) + \varepsilon_{||}\sin^2\vartheta(z)]^2} + \frac{\chi_a B^2}{\mu_0}\sin(\vartheta(z)+\psi)\cos(\vartheta(z)+\psi)\right] dz \qquad (3)$$

Since this torque causes the boundary orientation of the director different from the minimal-energy direction corresponding to nematics-substrate interaction, the boundary value of ϑ should be modelled by a reasonably chosen function, e.g. a polynomial of sufficiently large order [10–13,16,17]

$$\vartheta(0) = \vartheta(d) = \Theta(T_b) \qquad (4)$$

In particular case of strong anchoring this function is constant: $\vartheta(0) = \vartheta(d) = \vartheta_0$. In this work the third order polynomial is assumed [16,17]

$$\vartheta(0) = \vartheta(d) = \Theta(T_b) \cong \Theta_0 + \Theta_1 T_b + \Theta_2 T_b^2 + \Theta_3 T_b^3 \qquad (5)$$

Hence every stationary state of planar deformation of the layer can be characterised by a solution $\vartheta = \vartheta(z)$ and ε_e of Eq. (1) and φ from formula (2), with ϑ satisfying boundary condition (5), (4), (3) and corresponding to the values of material parameters $K_{11}, K_{33}, \chi_a, \Theta_0, \Theta_1, \Theta_2, \Theta_3, \varepsilon_\perp, \varepsilon_{||}, n_\perp, n_{||}$ and applied external forces $U, (B, \psi)$. On the other hand both the effective electric permittivity ε_e and the phase shift φ can be measured in experiment. The electric permittivities and refraction indices can be measured in separate experiments, as described above.

DETERMINATION OF NEMATICS MATERIAL CONSTANTS

Every characteristics of a nematics cell as a birefringence system, i.e. the dependence $\varphi = \varphi(U, B, \psi)$, contains the information on material constants $K_{11}, K_{33}, \chi_a, \Theta_0, \Theta_1, \Theta_2, \Theta_3, \varepsilon_\perp, \varepsilon_{||}, n_\perp, n_{||}$. Let $p = (K_{11}, K_{33}, \chi_a, \Theta_0, \Theta_1, \Theta_2, \Theta_3)$ denote the set of unknown material parameters. For any sequence of measurements $\left(\varphi_e(U^i, B^i, \psi^i)\right)_{i=1}^n$ one can calculate a corresponding sequence of values of light phase shift $\left(\varphi_c(U^i, B^i, \psi^i; p)\right)_{i=1}^n$. These two sequences can be compared by the following similarity functional [14,16,17]:

$$S(p) = \left\{ \frac{1}{n} \sum_{i=1}^{n} \left[\frac{\varphi_e(U^i, B^i, \psi^i) - \varphi_c(U^i, B^i, \psi^i; p)}{\varphi_e(U^i, B^i, \psi^i)} \right]^2 \right\}^{\frac{1}{2}} \tag{6}$$

which has the unknown material parameters p as arguments.

Now the inverse problem [14–17] can be posed: having the results of experiment $\left(\varphi_e(U^i, B^i, \psi^i)\right)_{i=1}^{n}$ given find the set of unknown parameters p by minimising this functional approximately.

RESULT OF EXPERIMENT

The optical response of the cell to a normally incident light (the phase shift between ordinary and extraordinary light ray) was measured as a sample physical quantity. The magnetic and electric characteristics of cells (i.e.

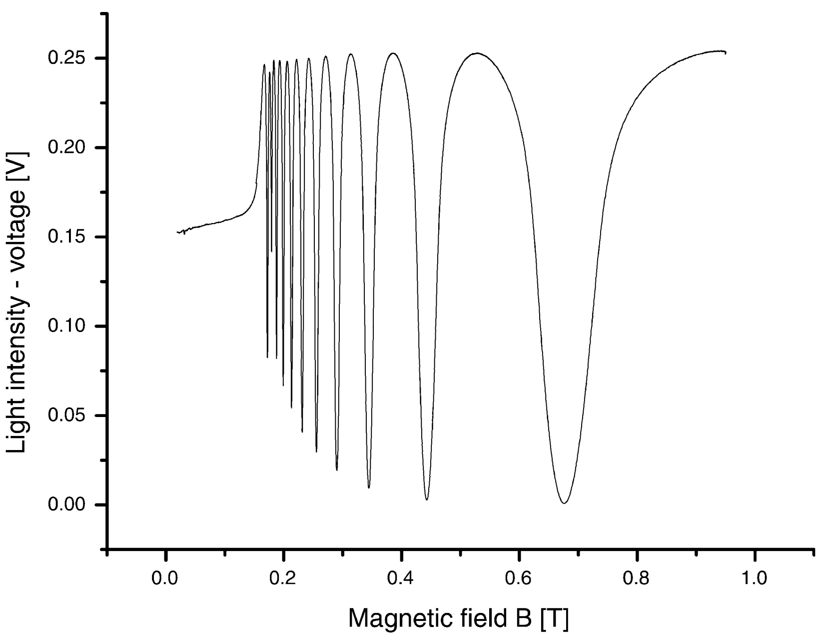

FIGURE 3 Intensity of a normally incident light (expressed as an equivalent voltage) as a function of a magnetic field, recorded for the 12th fringe corresponding to the cell thickness 42.3 μm, at the temperature 23.7°C.

the dependence of the phase shift on a magnetic field and a voltage applied to the cover electrodes) were recorded. The characteristics were treated in computations as functions of unknown material parameters as arguments. The magnitudes of the parameters were found by a non-linear least-squares fitting of the calculated characteristics to the measured ones, as described above.

The wedge nematics cell described above, with the covers coated with the polyimide MK8 and filled with PCB, was investigated by applying a magnetic field and a voltage. The measurements of the dependence of the intensity of normally incident light, transmitted through the cell, on external fields were performed in the system briefly described above. The light intensity as a function of a magnetic field B, perpendicular to the cell boundaries ($\psi = 0$), was recorded for the three interference fringes of order 12 (Fig. 3), 17 and 27 and the equivalent flat cell thickness 42.3 μm, 60.2 μm and 94.5 μm, at the temperature 23.7°C measured with

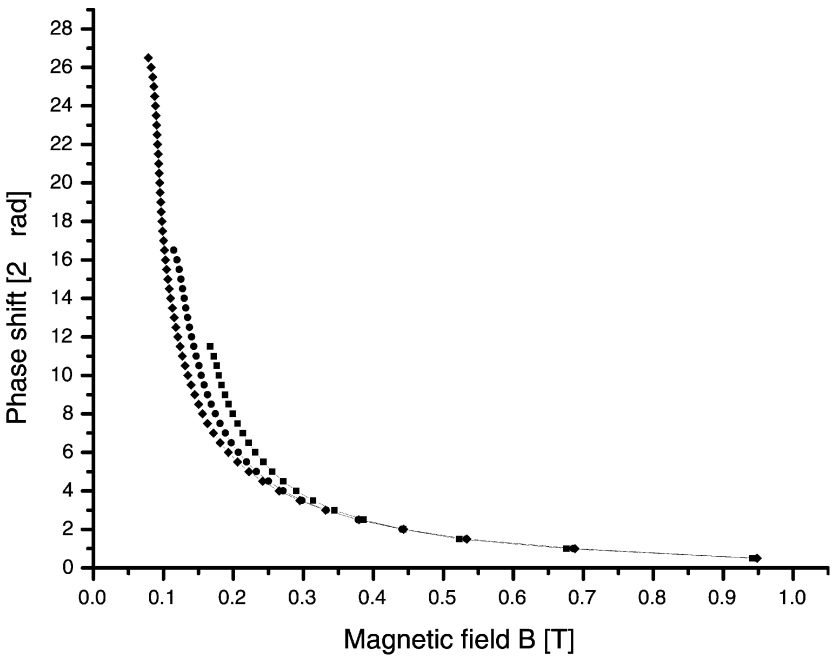

FIGURE 4 Phase shift between ordinary and extraordinary ray of normally incident light (in 2π rad units) as a function of a magnetic field B (in Tesla) acting perpendicularly to the nematics layer ($\psi = 0$), recorded for the 12th, 17th and 27th fringe, corresponding to the cell thickness 42.3 μm, 60.2 μm, 94.5 μm, at the temperature 23.7°C.

the accuracy within 0.1°C. The light intensity as a function of a voltage U was recorded for the same interference fringes at the same temperature by using the sinusoidal low-frequency (1.5 kHz) voltage. The magnetic $\varphi = \varphi(B, \psi)$ and the electric $\varphi = \varphi(U)$ optical characteristics were determined from these experimental functions by taking into account that each of their extremes corresponds to the phase shift equal to the integer multiplicity of π radians (Fig. 4). In this way the measurement of the phase shift was reduced to the precise determination of the magnitudes of the magnetic or electric field (abscissas) corresponding to the subsequent extremes (ordinates), as in Figure 3.

The electric permittivities and the refractive indices for red light ($\lambda = 0.6328\,\mu m$) were determined by smoothing and interpolating the experimental data from independent measurements and at the temperature 23.7°C were found to be as follows: $\varepsilon_{||} = 18.85$, $\varepsilon_{\perp} = 6.56$, $n_o = 1.5388$, $n_e = 1.7178$.

The inverse problem was solved for the data from the three magnetic characteristics. The minimisation of functional (6) was done toward achieving the best-possible fitting of the measured characteristics by the calculated ones with the same elastic constants K_{11}, K_{33} and the anisotropy of the diamagnetic susceptibility χ_a for all characteristics involved. The coefficients $\mathbf{\Theta}_0, \mathbf{\Theta}_1, \mathbf{\Theta}_2, \mathbf{\Theta}_3$ of a polynomial approximating the nematics-substrate coupling were determined for each set of data (for

TABLE 1 The Results of Measurements and Calculations. The Magnitudes of the Material Constants of PCB at the Temperature 23.7°C = T_{NI} − 10.8°C, Obtained as the Solution of the Inverse Problem, and the Corresponding Values of the Similarity Functional for the Six Analysed Characteristics (i.e. the Three Magnetic and The Three Electric, Corresponding to the 12th, 17th and 27th fringe). The Other Part of the Solution, i.e. The Characteristics of the Nematics-Substrate Coupling, is Presented in Figure 5.

Splay elastic constant K_{11}	Bend elastic constant K_{33}	Anisotropy of diamagnetic susceptibility χ_a
5.95 pN	9.18 pN	$1.53 \cdot 10^{-6}$
Fringe order and kind of characteristics	Equivalent cell thickness d	value of similarity funcational
12 m	42.3 μm	0.009
17 m	60.2 μm	0.007
27 m	94.5 μm	0.008
12 e	42.3 μm	0.008
17 e	60.2 μm	0.016
27 e	94.5 μm	0.023

six characteristics) separately to verify accuracy of the procedure. The results referring to the bulk elastic constants and the anisotropy of diamagnetic susceptibility and the final approximation errors are collected in Table 1. The refined fit parameters, i.e. the values of the similarity functional (6), are about 1%, which is the level of measurement error. The magnitudes of material constants for PCB, presented in Table 1, are in good agreement with those obtained in distinct measurements [18,19]. The polynomials $\vartheta(0) = \vartheta(d) = \Theta(T_b)$, obtained in all cases, were quite similar each other as it is illustrated in Figure 5.

The input data for the inverse problem, i.e. the magnetic characteristics $\varphi = \varphi(B, \psi)$ recorded for the equivalent cell of thickness 42.3 μm, 60.2 μm and 94.5 μm, are presented in Figure 4. The characteristics, calculated as corresponding to the solution of the inverse problem (K_{11}, K_{33}, χ_a and the

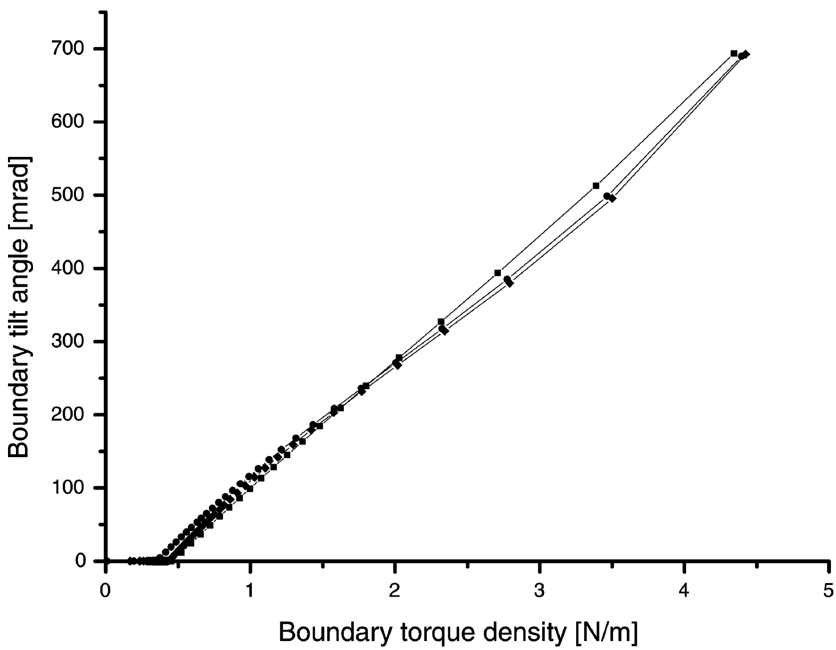

FIGURE 5 Boundary tilt angle as a function of a torque density $\vartheta(0) = \vartheta(d) = \Theta(T_b)$ i.e. the characterisation of the coupling between the nematics (PCB) and the substrate material (polyimide MK8) at the boundaries of the cell. The functions were obtained as the solutions of the inverse problem for the 12th, 17th and 27th fringe, corresponding to the cell thickness 42.3 μm, 60.2 μm, 94.5 μm, at the temperature 23.7°C, with the magnetic characteristics (presented in Figure 4) as the input data.

polynomials $\vartheta(0) = \vartheta(d) = \Theta(T_b)$), differ negligibly from the measured ones (the differences are invisible in the scale of figure). The functions $\Theta(T_b)$ (5), describing the dependence of the boundary tilt angle $\vartheta(0) = \vartheta(d)$ on the torque density T_b calculated according to formula (3) for the three magnetic characteristics, are shown in Figure 5. Each of them was modelled by two third-order polynomial, one for the values of the torque density from 0 till about 0.5 μN/m and the other for larger values, since one polynomial was not sufficient for approximating this function precisely. The values of the fit parameters, given in Table 1, were calculated for this approximation. The function $\vartheta(0) = \vartheta(d) = \Theta(T_b)$, describing the coupling between the nematics PCB and the substrate MK8, combined of two polynomials for the equivalent cell of thickness 60.2 μm, is shown in Figure 6 (for better view only the first part of the curve is presented). It is evident from Figures 5 and 6 that the anchoring is weak, with the anchoring direction equal to zero (within the accuracy of this analysis).

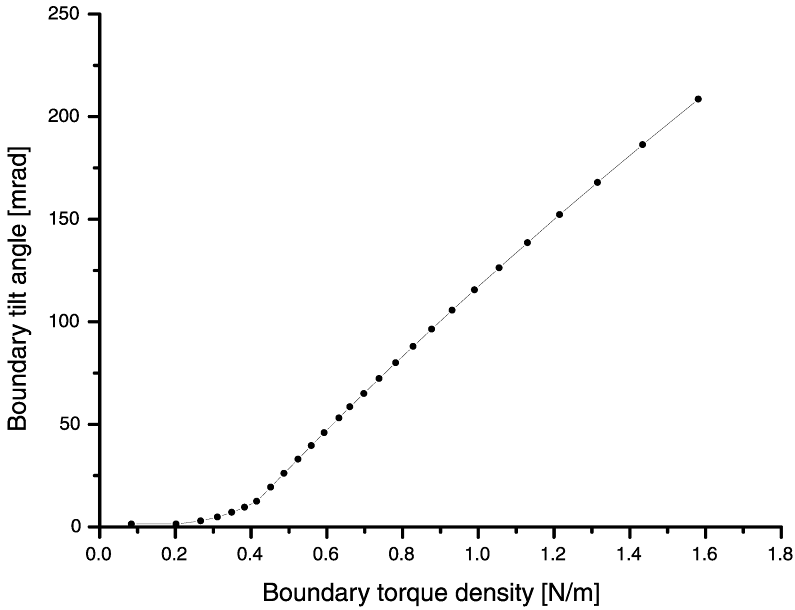

FIGURE 6 Boundary tilt angle as a function of a torque density $\vartheta(0) = \vartheta(d) = \Theta(T_b)$ for the nematics PCB - the substrate polyimide MK8 system, combined of two third-order polynomials as the solution of the inverse problem for the 17th fringe, corresponding to the cell thickness 60.2 μm at the temperature 23.7°C. Only the first part of the curve is shown (to compare with the whole curve in Figure 5).

CONCLUSION

A wedge cell of planar nematics alignment enables measurements of the optical or dielectric response (i.e. the effective electric permittivity or effective birefringence) to external magnetic or electric fields, substituting a set of flat-parallel cells of different thickness and with the same coating surface treatment. The thickness of equivalent flat cells may be precisely determined irrespectively of possible bend of cell covers, what makes the measurements be more precise.

The experimental characteristics of the nematics cell can be used as input data for inverse problems of determining the unknown magnitudes of bulk material parameters. The method presented above makes possible the quantitative determination of the characteristics of nematics-substrate interaction simultaneously with the determination of the nematics constants, both in the case of weak anchoring and in the case of strong anchoring. For obtaining reliable results it is necessary to take into account few different characteristics measured in a wide range of voltages and magnetic fields.

The combination of the method described above with a wedge cell as measurement device results in a tool for reliable repeatable determination of nematics constants and nematics-substrate coupling.

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